Organo ufficiale
dell'Area Oli e Grassi di Innovhub
Stazioni Sperimentali per l'Industria
Azienda Speciale
della Camera di Commercio di Milano

RISG

APRILE/GIUGNO 2017
ISSN 0035-6808 RISGARD 94 (2) 65-136 (2017)
Poste Italiane S.p.a. - Spedizione in Abbonamento Postale - 70%
Finito di stampare nel mese di Giugno 2017

94° ANNO

LA RIVISTA ITALIANA DELLE SOSTANZE GRASSE

2 2017

INNOVHUB
STAZIONI SPERIMENTALI
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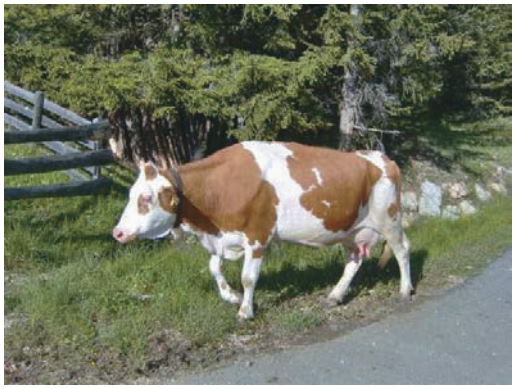

Il Settore Qualità Genuinità Micronutrienti e Sicurezza Alimentare svolge attività analitica nell'area della caratterizzazione delle *sostanze grasse* in generale e in particolare per la valutazione dei principali parametri di qualità e genuinità degli OLI di *oliva* e di *sansa* e *oliva* secondo le normative UE e COI vigenti e i diversi standard internazionali.

In particolare il settore è specializzato nella determinazione quali/quantitativa di composti fondamentali per la valutazione dello *stato di ossidazione*, della *shelf-life*, degli *antiossidanti naturali*, degli *indici di genuinità* e di *qualità nutrizionali*, dei *micronutrienti* quali le *vitamine* liposolubili e idrosolubili.

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Partecipa come membro attivo ai gruppi di lavoro per lo sviluppo delle normative nazionali e internazionali (COI, UE, CODEX, UNI).

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APRILE/GIUGNO 2017 - ANNO XCIV

RISG
LA RIVISTA ITALIANA DELLE SOSTANZE GRASSE

ORGANO UFFICIALE DELL'AREA OLI E GRASSI DI INNOVHUB
STAZIONI SPERIMENTALI PER L'INDUSTRIA
AZIENDA SPECIALE DELLA CAMERA DI COMMERCIO DI MILANO

Sommario

GRAFICA, IMPAGINAZIONE E STAMPA

Grafiche Parole Nuove srl
Via Garibaldi 58 - Brugherio

DIRETTORE RESPONSABILE: M. SURDI
REDAZIONE: F. PAPARELLA

REDAZIONE
ABBONAMENTI
franca.paparella@mi.camcom.it

Autorizzazione del Tribunale di Milano 28.9.1948, N. 591
Pubblicità inferiore al 70%

DIREZIONE E REDAZIONE:
Via Giuseppe Colombo 79 – 20133 Milano

Sito web: www.innovhub-ssi.it

Abbonamenti 2017: annuo Italia € 100,00 – annuo estero € 200,00
Numero singolo € 30,00

IMPACT FACTOR 2015: 0,417

Indexed and Abstracted in:
- **Thomson Scientific Service:** Science Citation Index Expanded (SciSearch®), Journal Citation/Science Edition, Current Contents®/ Clinical Medicine
- **Chemical Abstracts**
- **Elsevier Bibliographic Databases:** SCOPUS
- **FSTA – Food Science and Technology Abstract** (IFIS Publishing – UK)

La RIVISTA ITALIANA DELLE SOSTANZE GRASSE è l'organo ufficiale dell'Area Oli e Grassi di Innovhub - Stazioni Sperimentali per l'Industria - Azienda Speciale della Camera di Commercio di Milano. Ha periodicità trimestrale e la scientificità dei contenuti è garantita da un Comitato Internazionale di Referee.

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Determinazione dei prodotti fitosanitari nell'olio extra vergine di oliva: validazione e indagine conoscitiva

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Determination of pesticides in extra virgin olive oil: validation and survey
A new analytical method was developed for the determination multiresidual of plant protection products, belonging to different chemical categories, using the technique UHPLC/MS-MS triple quadrupole with H-ESI-II interface. Validation experiments were conducted following the guidelines of the SANCO 12571/2013 protocol [1] to determine the lowest calibration level (LCL), the linearity of the instrumental response (R^2), the recovery, the repeatability (r) and percent standard deviation for repeatability (RSDr%). As regards recoveries, 83% of the principles showed values comprised in reference range (70% - 120%). The principles that have not complied with this reference range showed yield lower values, only three of them, yielded higher values (Table III). 97% of the principles showed a residual value relative to the calibration curve in matrix ≤ 20%, 99% of the principles showed RSDr values ≤ 20%, 91% of the residues possessed of at least 2 transitions in the spectrum fragmentation. All the principles analyzed showed LCL values ≤ maximum residue limits set by the EC Regulation 396/2005 [2]. Once validated, the analytical method was used for the analysis of 23 samples of extra virgin olive oil, collected during the year 2015/2016 in the large national distribution in order to conduct an analysis of survey type.

Una nuova metodica analitica è stata messa a punto per la determinazione multiresiduale di prodotti fitosanitari, appartenenti a diverse categorie chimiche, utilizzando la tecnica UHPLC/MS-MS triplo quadrupolo con interfaccia H-ESI-II. Esperimenti di validazione sono stati condotti seguendo le linee guida del protocollo SANCO 12571/2013 [1] per determinare il valore di lowest calibration level (LCL), la linearità della risposta strumentale (R^2), il recupero, la ripetibilità (r) e la deviazione standard percentuale della ripetibilità (RSDr %). Per quanto riguarda i recuperi, l'83% dei principi ha mostrato valori compresi nell'intervallo di riferimento (70% - 120%). I principi che non hanno rispettato questo range di riferimento tendenzialmente hanno mostrato valori di resa inferiori, solo tre di essi, valori di resa superiore (Tabella III). Il 97% dei principi ha mostrato un valore dei residui relativi alla curva di calibrazione in matrice ≤ 20%, il 99% dei principi ha mostrato valori di RSDr ≤ 20%, il 91% dei residui era in possesso di almeno 2 transizioni nello spettro di frammentazione. Tutti i principi analizzati hanno mostrato valori di LCL ≤ ai limiti massimi per i residui fissati dal Regolamento CE 396/2005 [2]. Una volta validato, il metodo analitico è stato utilizzato per l'analisi di 23 campioni di olio extravergine di oliva, prelevati nel corso dell'anno 2015/2016 nella grande distribuzione nazionale al fine di condurre un'analisi di tipo conoscitivo.

INTRODUZIONE

La necessità di garantire produzioni agricole quantitativamente e qualitativamente elevate e nello stesso tempo di difendere le colture da attacchi di parassiti, insetti o funghi, comporta ancora oggi un largo impiego di prodotti fitosanitari [4 - 6]. Si tratta di molecole che a causa delle loro caratteristiche chimico-fisiche possono esplicare effetti negativi sull'uomo e sull'ambiente. Solo una piccola parte dei prodotti che vengono utilizzati nelle colture raggiungono gli organismi bersaglio; inevitabilmente una parte di questi si depositano sul terreno o sui prodotti agricoli in cui, a seconda delle loro caratteristiche, possono accumularsi. Quantità piccole possono essere riscontrate anche negli alimenti e quindi raggiungere il consumatore, che deve essere tutelato da eventuali effetti tossici [7–11, 19-21]. La normativa vigente prescrive controlli sempre più rigorosi sull'uso corretto dei prodotti fitosanitari nelle produzioni agricole e sui relativi residui negli alimenti. Per ogni principio attivo, a seguito della rispettiva autorizzazione commerciale in base al Regolamento CE 1107/2009 [15], il Regolamento CE 396/2005 [2] stabilisce un valore di MRL (maximum residue level) ovvero "la concentrazione massima ammissibile di residuo in alimenti assicurando la minima esposizione possibile ai consumatori". Questi limiti vengono fissati anche sulla base delle GAP (good agricultural practices), ossia sul rispetto delle condizioni di impiego (dosi, numero di trattamenti, intervallo di sicurezza). I valori di MRLs sono diversi per ciascun principio attivo e non tutti i principi utilizzati in agricoltura sono regolamentati; in quest'ultimo caso viene assunto un valore MRL di riferimento pari o inferiore a 0,01 mg/kg. Per l'olio di oliva il regolamento sopracitato non stabilisce un valore di MRL nel prodotto finito ma sul frutto (oliva). Per ottenere il corrispondente limite sull'olio è necessario applicare un fattore di trasformazione pari a 5 (considerando una resa di estrazione dell'olio di oliva del 20%) come stabilito dal Regolamento UE 788/2012 [3].

Per quanto riguarda l'olio biologico, esso è regolamentato dal Decreto Ministeriale D.M. 309 del 2011 e dal suo Allegato I [13] che per quanto riguarda i limiti fa riferimento al Reg. CE 396/2005 per i principi presenti nell'allegato II del Reg. CE 889/2008 [14], mentre per gli altri principi autorizzati in agricoltura viene fissato un limite restrittivo pari a 0,01 mg/kg. Per tutti gli altri principi non autorizzati nell'agricoltura il regolamento di riferimento è sempre il Reg. CE 396/2005 [2].

La Relazione Annuale del Ministero della Salute riguardante il Controllo Ufficiale sui residui di prodotti fitosanitari negli alimenti, per l'anno 2014 [18] afferma che il 91,9% dei campioni di olio di oliva controllati è risultato privo di residui rilevabili, l'8,1% è risultato con residui inferiori al limite di legge.

In passato nel laboratorio si era già affrontato uno studio similare in maniera più contenuta utilizzando la tecnica dall'HPLC-MS/MS con interfaccia APCI [12].

MATERIALI E METODI

MATERIALI

- 23 campioni di olio extravergine di oliva prelevati nella grande distribuzione in tutta Italia, nel corso dell'anno 2015/2016, di diversa origine dichiarata in etichetta, in particolare: 9 di origine italiana, di cui 3 biologici, 11 di origine europea e 3 di origine extra europea (Tabella I).
- 1 campione bianco di olio extravergine di oliva biologico, preanalizzato per il contenuto dei residui.
- Bilancia analitica di precisione (± 0,001 g).
- Matracci ambrati tarati di classe A da 10 ml e da 20 ml
- Pipette elettroniche di precisione
- Bagnomaria riscaldante con temperatura di controllo a 30°C e flusso di azoto
- Vortex per provette
- Bagno di estrazione ad ultrasuoni
- Pipette tipo pasteur in vetro
- Centrifuga da banco con capacità di rotazione fino a 5000 rpm
- Filtri per siringa in PVDF da 0,22 µm diametro 13 mm
- Colonna per UHPLC Hypersil Gold 50 mm × 2.1 mm 1,9 µm (ThermoFisher, USA)
- Vials ambrate per autocampionatore da 2 ml con tappo a vite e setto in PTFE/silicone
- Provette in plastica da 15 ml con tappo a vite, monouso.

REAGENTI

- Acqua LC-MS, metanolo LC-MS, LC-MS e acetonitrile tipo pestanal per l'analisi dei residui (Sigma Aldrich Steihnem, DE)
- Ammonio formiato purezza > 99% (Fluka, DE)
- Soluzioni di miscele di prodotti fitosanitari (LGC STANDARDS Bury, UK) a concentrazione di 100 ng/µl in acetonitrile (stock solutions) con circa 20 principi ciascuna
- Soluzione di standard interno deuterato Malathion D-6 (LGC STANDARDS Bury, UK), a concentrazione di 100 ng/µl in acetonitrile.

TUNING SOLUTIONS

Le soluzioni per il tuning dello strumento sono state preparate prelevando 100 µl dalle singole stock solutions (100 ng/µl) e successivamente diluite con metanolo in matraccio tarato da 10 ml. Le soluzioni ottenute avevano una concentrazione di 1 µg/ml. Le soluzioni di tuning sono state conservate in frigorifero a +4°C per 6 mesi.

WORKING SOLUTION

La soluzione standard di lavoro rappresenta la base per la costruzione sia della curva di calibrazione in matrice sia per quella in solvente. È stata preparata prelevando 100 µl da ogni singola stock solution (100 ng/µl) e diluita in acetonitrile pestanal, in un matraccio

Tabella I - Elenco dei campioni di olio extra vergine di oliva analizzati

Campioni	Origine dichiarata	Altre caratteristiche
1	Italia	n.d
2	Italia	Biologico
3	Unione europea	n.d
4	Italia	n.d
5	Unione Europea	n.d
6	Italia	n.d
7	Unione Europea	n.d
8	Italia	n.d
9	Unione Europea	n.d
10	Ue-Extra Ue	n.d
11	Unione Europea	n.d
12	Unione Europea	n.d
13	Ue-Extra Ue	n.d
14	Italia	Biologico
15	Unione Europea	n.d
16	Unione Europea	n.d
17	Unione Europea	n.d
18	Unione Europea	n.d
19	Unione Europea	n.d
20	Ue-Extra Ue	n.d
21	Italia	Biologico
22	Italia	DOP Puglia
23	Italia	DOP Puglia

Tabella II - Condizioni operative del sistema UHPLC-MS/MS

Flusso UHPLC	300 µl/min
Sheat gas N2	40 u.a.
Auxiliary Gas N2	10 u.a.
Spray Voltage	3000 V (+) / 2100 V (-)
Temperatura Vaporizzatore	40°C
Temperatura Capillare	250°C
Fase mobile	Soluzione tampone di ammonio formiato 2 mM allo 0.05% di acido formico/metanolo 50/50 v/v

tarato da 10 ml, ottenendo una soluzione con concentrazione pari a 1 µg/ml. La soluzione è stata conservata in frigo a +4°C per 6 mesi.

PREPARAZIONE DELLA SOLUZIONE DI STANDARD INTERNO

50 µl sono stati prelevati dalla stock solution di Malathion D-6 (100 ng/µl) e diluiti in matraccio tarato da 20 ml con acetonitrile pestanal ottenendo una concentrazione di 0,25 µg/ml. La soluzione è stata conservata in frigorifero a +4°C per 6 mesi.

CURVA DI CALIBRAZIONE IN SOLVENTE

Dalla working solution (1 µg/ml) sono stati preparati dodici livelli di calibrazione in un intervallo compreso tra 0,0 µg/ml e 1,0 µg/ml, in acetonitrile pestanal, a ciascuno dei quali sono stati aggiunti 5 µl di standard interno Malathion-D6 (100 ng/µl). Ciascun livello aveva un volume finale di 1 ml. I singoli livelli erano stabili per tre mesi dalla data di preparazione e conservati in frigorifero a +4°C. 5 µl di ciascuna soluzione di calibrazione sono stati iniettati nel sistema UHPLC/MS-MS in triplicato. Dalla curva di calibrazione in solvente sono state ricavate le equazioni di linearità, i valori R^2, dei residui e i valori di LCL in solvente.

CURVA DI CALIBRAZIONE IN MATRICE

La calibrazione in matrice è stata utilizzata per compensare gli effetti dovuti alla matrice sulla ionizzazione in spettrometria di massa e per effettuare il processo di quantificazione. È stata preparata utilizzando un olio extravergine di oliva, precedentemente analizzato, in cui non è stata rilevata la presenza di prodotti fitosanitari (campione bianco). Sono state eseguite 12 estrazioni indipendenti ciascuna delle quali utilizzata per costruire un differente livello di concentrazione della curva di calibrazione. L'aggiunta dello standard interno (Malathion D6 100 ng/µl) e degli standard esterni presenti nella working solution (1 µg/ml) è stata fatta al termine delle estrazioni del campione dopo la fase di evaporazione a temperatura ambiente sotto flusso di azoto seguendo lo stesso schema utilizzato per costruire la curva in solvente. 5 µl di ciascuna soluzione sono stati iniettati nel sistema UHPLC/MS-MS in triplicato. Tale soluzione era stabile in frigorifero a +4°C per 2 mesi. Dalla curva in matrice sono state ricavate le equazioni di linearità, i valori R^2, dei residui e i valori ipotetici di LCL in matrice (Tabella III).

ACCURATEZZA E RIPETIBILITÀ

Al fine di poter confermare il valore di LCL ipotizzato per ciascun residuo sono stati pesati 5 g di un campione bianco e ad esso sono stati aggiunti 250 µl della working solution (1 ug/ml), 1 ml della soluzione di standard interno Malathion D6 (0,25 µg/ml) e 1 ml di acetonitrile pestanal. La soluzione ottenuta è stata miscelata su Vortex per 1 minuto, sonicata nel bagno ad ultrasuoni per 10 minuti e centrifugata a 5000 rpm per 10 minuti. Il surnatante è stato trasferito in una provetta da 15 ml. Sono state effettuate altre due estrazioni ciascuna con 2 ml di acetonitrile e i surnatanti sono stati riuniti e filtrati su filtro a siringa in PVDF. Il filtrato è stato portato a secchezza su bagnomaria a temperatura non superiore a 30°C sotto flusso di azoto, diluito in 0,5 ml di acetonitrile pestanal, agitato su Vortex per 1 minuto, centrifugato per 5 minuti a 5000 rpm per isolare tracce di olio residuo. La soluzione è stata trasferita in microvials da 2 ml e iniettata nel sistema UHPLC/MS/MS in triplicato. Il campione così preparato aveva una concentrazione di 0,05 mg/kg di ciascun residuo fitosanitario. Con questo procedimento sono stati preparati 8 estratti indipendenti e da questa fase di validazione si sono confermati i valori di LCL in matrice e ottenuti i valori di accuratezza, di ripetibilità (r) e di RSDr % (Tabella III).

PREPARAZIONE DEI CAMPIONI DA ANALIZZARE

La preparazione dei campioni di analisi è stata condotta con lo stesso metodo descritto sopra senza ag-

TABELLA III - Parametri di ionizzazione e di frammentazione utilizzati. Sono riportati: il principio attivo, la categoria, la classe di appartenenza, la forma dello ione pseudomolecolare, il valore m/z dello ione parentale, il valore della S-lens, l'energia di collisione (CE%) espressa in Volt, i valori m/z degli ioni frammento e parametri di validazione relativi alla curva in matrice R^2, residui, LCL (mg/kg), Recupero %, r (mg/kg) ripetibilità, RDSr % (deviazione standard percentuale della ripetibilità)

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R^2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
	Md6 (IS)			$[M+H^+]^+$	337.0	85	31	131.0	-	-	-	-	-	-
					337.0		5	291.0						
1	Acephate	IN	Organofosforato	$[M+H^+]^+$	184.0	74	20	113.0	0,99	17	0,00100	106	0,004	2
					184.0		5	142.9						
2	Acybenzolar-s-methyl	PA	Benzotiadiazolo	$[M+H^+]^+$	211.0	135	20	91.1	0,97	20	0,00500	70	0,009	7
					211.0		30	136.1						
3	Alachlor	HB	Cloroacetanilide	$[M+H^+]^+$	270.1	92	20	162.2	1,00	9	0,00025	93	0,001	1
					270.1		11	238.2						
4	Aldicarb	NE, IN, AC	Carbammato	$[M+Na^+]^+$	213.0	122	16	89.0	0,93	8	0,05000	85	0,029	20
					213.0		11	116.0						
5	Aldicarb sulfone	NE, IN, AC metabolita	Carbammato	$[M+H^+]^+$	223.1	77	13	86.1	0,99	15	0,00050	87	0,002	1
					223.1		10	148.0						
6	Aldicarb sulfoxide	NE, IN, AC metabolita	Carbammato	$[M+H^+]^+$	207.0	70	16	89.0	0,99	12	0,00250	95	0,008	5
					207.0		10	132.0						
7	Ametryn	HB	Triazinico	$[M+H^+]^+$	228.1	95	36	68.1	0,99	10	0,00050	70	0,007	6
					228.1		18	186.1						
8	Amitraz	AC,IN	Formamidico	$[M+H^+]^+$	294.4	79	-	294.4	0,97	16	0,00250	15	0,004	16
9	Atrazine	HB	Triazinico	$[M+H^+]^+$	216.0	95	28	104.0	0,98	20	0,00150	70	0,008	7
					216.0		18	174.1						
10	Azadirachtin	IN	Piretrina	$[M-OH]^+$	703.2	163	14	567.3	0,96	26	0,02500	85	0,029	20
					703.2		17	585.2						
11	Azinphos ethyl	IN,AC	Organotiofosfato	$[M-C4H10+2H]^+$	289.0	94	21	233.1	0,98	15	0,00100	79	0,012	9
12	Azinphos methyl	IN,AC	Organotiofosfato	$[M+H^+]^+$	261.0	90	18	125.0	0,99	18	0,00250	76	0,008	6
					261.0		15	167.0						
13	Azoxystrobin	FU	Metossiacrilato	$[M+H^+]^+$	404.1	116	31	329.0	0,99	16	0,00025	89	0,006	4
					404.1		14	372.0						
14	Benalaxyl	FU	Anilide	$[M+H^+]^+$	326.2	108	39	91.1	0,99	1	0,00025	77	0,001	1
					326.2		23	148.1						
15	Bentazone (-)	HB	Benzotiadiazina	[M-H]-	239.0	112	29	132.1	0,99	12	0,00150	78	0,013	10
					239.0		22	197.1						
16	Boscalid	FU	Anilide	$[M+H^+]^+$	343.1	156	30	271.2	0,99	10	0,00025	70	0,002	2
					343.1		17	307.2						
17	Bromophos ethyl	IN	Organotiofosfato	$[M+H^+]^+$	393.0	100	18	155.0	0,98	12	0,00150	103	0,001	1
					393.0		52	172.0						
18	Bromophos methyl	IN	Organotiofosfato	$[M+H^+]^+$	365.0	100	29	99.1	0,98	9	0,01000	107	0,002	1
					365.0		37	174.0						
19	Buprofezin	IN	Tiodiazinone	$[M+H^+]^+$	306.3	80	16	116.0	0,99	9	0,00025	56	0,003	3
					306.3		10	201.0						
20	Cadusafos	IN,NE	Organotiofosfato	$[M+H^+]^+$	271.1	76	21	131.0	1,00	13	0,00025	64	0,003	3
					271.1		13	159.1						
21	Carbaryl	IN,PG	Carbammato	$[M+H^+]^+$	202.1	_	29	127.2	0,98	18	0,00500	95	0,006	4
					202.1		10	145.2						

Segue tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
22	Carbendazim	FU	Benzimidazolo	$[M+H^+]^+$	192.1	107	28	132.1	0,99	14	0,00100	57	0,009	9
					192.1		19	160.1						
23	Carbofuran	IN,NE,AC	Carbammato	$[M+H^+]^+$	222.1	92	21	123.1	0,99	3	0,00150	92	0,006	4
					222.1		12	165.1						
24	Chlorfenvinfos	IN	Organofosforato	$[M]^+$	359.0	110	39	170.0	0,98	13	0,00050	102	0,002	1
					359.0		20	205.0						
25	Chlorpyrifos ethyl	IN,AC	Organoclorurato	$[M]^+$	350.0	110	20	198.0	0,97	20	0,00100	51	0,01	12
					350.0		10	322.0						
26	Chlorpyrifos methyl	IN,AC	Organoclorurato	$[M+H^+]^+$	321.9	100	19	125.0	0,97	12	0,00250	70	0,014	12
					321.9		16	289.9						
27	Cyfluthrin	IN,AC	Piretroide	$[M+H^+]^+$	434.1	173	24	220.0	0,99	16	0,00150	100	0,015	9
					434.1		20	395.0						
28	Cyproconazole	FU	Conazolo	$[M+H^+]^+$	292.1	120	20	70.1	0,99	20	0,00025	99	0,016	10
					292.1	64	32	125.0						
29	Daminozide	PG	Idrazide	$[M^+]^+$	160.1	137	26	105.1	0,98	20	0,00250	88	0,008	6
					160.1		21	132.1						
30	Deltamethrin	IN	Piretroide	$[M+NH_4^+]^+$	523.1	93	33	181.2	0,97	10	0,01000	59	0,009	9
					523.1		17	180.8						
31	Demethon-s-methyl	IN,AC	Organotiofosfato	$[M+H^+]^+$	231.1	94	21	111.2	-	-	-	-	-	-
					231.1		22	129.0						
32	Demethon-s-methyl sulfone	IN	Organotiofosfato	$[M+H^+]^+$	263.1	106	30	109.1	0,99	15	0,00025	78	0,003	2
					263.1		22	125.1						
33	Demethon-s-methyl sulfoxide	IN metabolita	Organotiofosfato	$[M+H^+]^+$	246.9	97	14	169.1	0,99	11	0,00025	106	0,003	2
34	Diazinon	IN,AC	Organotiofosfato	$[M+H^+]^+$	305.0	100	19	153.0	0,99	19	0,00025	75	0,003	3
					305.0		22	169.0						
35	Dichlorvos	IN,AC	Organofosforato	$[M+H^+]^+$	221.0	80	24	79.1	0,99	2	0,00500	70	0,03	25
36	Dicofol	AC	Oragnoclorurato	$[M+H^+]^+$	369.0	101	35	205.9	0,99	15	0,00150	77	0,004	3
					369.0		36	208.0						
37	Diethofencarb	FU	Carbalinato	$[M+H^+]^+$	268.2	83	31	124.0	0,99	9	0,00100	107	0,005	3
					268.2		17	180.1						
38	Difenoconazole	FU	Conazolo	$[M]^+$	406.2	81	55	111.0	0,99	14	0,00025	74	0,002	1
					406.2		25	251.0						
39	Diflubenzuron	IN	Benzofenilureico	$[M+H^+]^+$	311.1	90	31	141.1	0,99	14	0,00150	76	0,002	1
					311.1		14	158.1						
40	Diflufenican	HB	Anilide	$[M+H^+]^+$	395.0	_	32	246.0	0,99	20	0,00050	71	0,006	5
					395.0		23	266.0						
41	Dimethoate	IN,AC	Oragnofosforato	$[M+H^+]^+$	230.1	95	20	125.0	0,99	6	0,00050	80	0,004	3
					230.1		10	199.0						
42	Diquat	HB,DE	Ammina quaternaria	$[M+H^+]^+$	183.0	-	31	130.0	0,95	1	0,00100	-	-	-
					275.1		15	199.1						
43	Disulfoton	IN	Organotiofosfato	[M+H+]+	275.1	94	37	125.1	0,93	20	0,05000	76	0,027	20
					275.1		15	199.1						

Segue Tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
44	Disulfoton sulfone	IN metabolita	Organotiofosfato	$[M^+]^+$	307.1	155	33	97.0	0,99	17	0,00050	82	0,005	3
					307.1		15	171.1						
45	Disulfoton sulfoxide	IN metabolita	Organotiofosfato	$[M+H^+]^+$	291.0	85	22	157.0	0,99	1	0,00050	111	0,005	3
					291.0		14	185.0						
46	Diuron	HB	Fenilureico	$[M+H^+]^+$	233.0	_	14	46.3	0,99	11	0,00100	76	0,004	3
					233.0		18	72.0						
47	DMST	nd	Fenilsufamide	$[M+H^+]^+$	215.1	87	28	106.0	0,99	8	0,00150	72	0,004	3
48	Endosulfan sulfate(-)	IN,AC metabolita	Organoclorurato ciclodiene	[M-H]-	421.0	100	72	80.0	0,99	14	0,00050	76	0,006	5
					421.0		51	97.0						
49	Endrin	IN	Oragnoclorurato ciclodiene	$[M+NH_4^+]^+$	398.9	110	20	127.0	0,99	19	0,00250	78	0,005	4
					398.9		25	240.8						
50	Ethion	IN,AC	Organotiofosfato	$[M+H^+]^+$	385.0	100	46	97.0	0,96	12	0,01000	72	0,004	3
					385.0		10	199.1						
51	Etoprophos	NE,IN	Organotiofosfato	$[M+H^+]^+$	243.1	100	19	131.0	0,99	6	0,00025	72	0,002	1
					243.1		14	173.1						
52	Famoxadone	FU	Dicarbossiamide	$[M+H^+]^+$	375.0	129	39	128.0	0,99	15	0,00025	63	0,017	16
					375.0		19	304.8						
53	Fenamidone	FU	Imidazolo	$[M+H^+]^+$	312.1	98	30	92.1	0,99	13	0,00025	153	0,023	9
					312.1		50	236.2						
54	Fenitrothion	IN,AC	Organotiofosfato	$[M+H^+]^+$	278.0	105	20	109.1	0,98	27	0,01000	117	0,018	9
					278.0		21	125.1						
55	Fenoxycarb	IN	Fenilureico	$[M+H^+]^+$	302.2	84	18	88.1	0,99	8	0,00500	70	0,003	2
					302.2		10	116.1						
56	Fenpropathrin	IN.AC	Piretroide	$[M+NH_4^+]^+$	367.0	201	20	125.0	0,94	25	0,05000	98	0,019	11
					367.0		20	350.0						
57	Fenthion	IN	Organotiofosfato	$[M+H^+]^+$	279.0	100	13	247.1	0,98	16	0,00025	71	0,003	3
58	Fenthion sulfone	IN	Organotiofosfato	$[M+H^+]^+$	311.0	172	21	125.0	0,97	10	0,00100	84	0,002	1
					311.0	_	15	217.0						
59	Fenthion sulfoxide	IN	Organotiofosfato	$[M+H^+]^+$	295.0	118	33	109.0	0,99	10	0,00100	94	0,005	3
					295.0		33	125.0						
60	Flucytrinate	IN	Piretroide	$[M+NH_4^+]^+$	469.3	_	30	412.0	0,97	15	0,00250	70	0,015	13
61	Folpet	FU	Ftalamide	$[M+NH_4^+]^+$	314.7	97	19	163.0	0,99	8	0,00050	82	0,001	1
					314.7		32	120.0						
62	Fonophos	IN	Organotiofosfato	$[M+H^+]^+$	247.0	90	19	109.1	0,99	20	0,00250	72	0,006	5
					247.0		8	137.1						
63	Formothion	IN,AC	Organotiofosfato	$[M+H^+]^+$	258.0	69	25	125.0	0,99	4	0,01000	98	0,013	8
					258.0		15	170.9						
64	Glyphosate	HB	Organofosforato	[M-H]-	168.0	52	41	79.1	0,96	7	0,00100	-	-	-
					168.0		15	81.1						
65	Haloxyfop-p	HB	ArilossiFenossiPropionico	$[M+H^+]^+$	362.1	106	26	288.2	0,99	14	0,00050	92	0,007	5
					362.1		18	316.1						
66	Haloxyfop-p ethoxyethyl	HB	ArilossiFenossiPropionico	$[M+H^+]^+$	434.2	118	28	288.1	0,99	7	0,00025	83	0,004	3
					434.2		19	316.2						

Segue Tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
67	Haloxyfop-p-methyl	HB	ArilossiFenossiPropionico	$[M+H^+]^+$	376.1	125	33	272.1	0,99	10	0,00025	81	0,002	2
					376.1		16	316.1						
68	Imazalil	FU	Conazolo	$[M+H^+]^+$	297.1	130	22	159.0	0,99	12	0,00025	71	0,01	8
					297.1		17	201.0						
69	Imidacloprid	IN	Neonicotinoide	$[M+H^+]^+$	256.0	91	20	175.2	0,98	20	0,00250	77	0,003	3
					256.0		16	209.2						
70	Indoxacarb	IN	Oxadiazina	$[M+H^+]^+$	528.1	144	28	150.0	0,99	14	0,00150	93	0,006	4
					528.1		41	203.0						
71	Iprovalicarb	FU	Carbammato Vanilamide	$[M+H^+]^+$	321.2	81	43	91.1	0,99	15	0,00025	185	0,006	2
					321.2		20	119.1						
72	Isofenphos	IN	Organofosforato	$[M+H^+]^+$	346.2	90	19	74.2	0,94	20	0,01000	70	0,005	4
					346.2		20	216.9						
73	Isoproturon	HB	Fenilureico	$[M+H^+]^+$	207.1	92	17	72.1	0,99	12	0,00025	79	0,001	1
					207.1		13	165.1						
74	Kresoxym methyl	FU	Strobilurina	$[M+H^+]^+$	314.2	68	17	116.0	0,98	18	0,00150	87	0,003	2
					314.2		14	222.0						
75	λ-Cyhalothrin	IN	Piretroide	$[M+NH_4^+]^+$	467.1	145	57	141.2	0,99	20	0,01000	70	0,013	11
					467.1		18	224.9						
76	Linuron	HB	Fenilureico	$[M+H^+]^+$	249.0	_	19	160.1	0,99	19	0,00025	72	0,009	7
					249.0		19	182.2						
77	Lufenuron	IN	Benzofenilureico	$[M+H^+]^+$	511.0	144	40	141.0	0,97	20	0,01000	53	0,013	15
					511.0		18	158.1						
78	Malaoxon	nd	Organofosforato	$[M+H^+]^+$	315.0	101	24	98.9	0,99	20	0,00025	92	0,007	5
					315.0		12	127.0						
79	Malathion	IN,AC	Organotiofosfato	$[M+H^+]^+$	331.0	100	24	99.0	0,99	11	0,00100	108	0,004	2
					331,0		12	127.0						
80	Mandipropamid	FU	Amide	$[M+H^+]^+$	412.1	119	34	125.0	0,99	16	0,00150	97	0,012	8
					412.1		14	328.1						
81	Mecarbam	IN,AC	Organotiofosfato	$[M+H^+]^+$	330.0	73	14	198.9	0,99	8	0,00250	90	0,003	2
					330.0		8	227.1						
82	Metalaxyl	FU	Acilamico	$[M+H^+]^+$	280.0	58	16	192.1	0,99	20	0,00025	89	0,004	2
					280.0		16	220.1						
83	Methacrifos	IN	Organotiofosfato	$[M+H^+]^+$	209.1	92	29	79.1	0,99	10	0,00150	73	0,009	7
					209.1		17	125.1						
84	Methidathion	IN,AC	Organotiofosfato	$[M+H^+]^+$	303.2	60	20	85.0	0,97	20	0,00500	93	0,012	8
					303.2		8	145.0						
85	Methoxyfenozide	IN	Diacilidrazina	[M-H]-	367.2	80	22	149.0	0,99	8	0,00050	79	0,004	3
86	Mevinphos	IN,AC	Organofosforato	$[M+H^+]^+$	225.1	84	30	109.1	-	-	-	-	-	-
					225.1		16	127.1	-	-	-	-	-	-
87	Monocrotophos	AC,IN	Oragnofosforato	$[M+H^+]^+$	224.1	172	12	98.1	0,99	20	0,00100	89	0,005	4
					224.1	62	17	127.0						
88	Novaluron	IN	Benzofenilureico	[M-H]-	491.0	108	20	305.1	0,99	15	0,00025	118	0,008	4
					491.0		15	471.1						

Segue Tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
89	Omethoate	IN,AC	Oragnofosforato	$[M+H^+]^+$	214.0	_	16	155.0	0,99	20	0,00050	86	0,005	3
					214.0		11	183.0						
90	Oxadiazon	HB	Oxadiazolone	$[M+H^+]^+$	345.1	115	31	185.0	0,99	15	0,00150	78	0,004	3
					345.1		19	219.9						
91	Oxadixyl	FU	Anilide	$[M+H^+]^+$	279.0	115	18	149.0	0,99	13	0,00025	88	0,005	4
					279.0		10	219.2						
92	Oxamyl	IN,NE	Carbammato	$[M^+]^+$	219.1	117	39	117.1	0,99	19	0,00025	85	0,008	6
					219.1		26	132.1						
93	Oxyfluorfen	HB	Difeniletere	$[M+H^+]^+$	362.1	125	46	140.0	0,99	13	0,00100	57	0,016	17
					362.1		27	237.0						
94	Paraoxon ethyl	PG	Organofosforato	$[M+H^+]^+$	276.2	90	36	94.1	0,98	6	0,00250	79	0,004	3
					276.2		23	220.1						
95	Paraoxon methyl	nd.	Orgnaofosforato	$[M+H^+]^+$	248.1	121	24	90.1	0,98	8	0,00050	79	0,008	6
					248.1		19	202.1						
96	Paraquat	HB	Ammina quaternaria	$[M+H^+]^+$	185.0	-	27	169.0	0,95	9	0,00100	-	-	-
					185.0		17	170.0						
97	Parathion ethyl	IN, AC	Organotiofosfato	$[M+H^+]^+$	292.1	100	35	94.0	0,98	25	0,01000	84	0,006	4
					292.1		13	236.1						
98	Parathion methyl	IN,RE	Organotiofosfato	$[M+H^+]^+$	264.0	116	20	125.0	0,95	13	0,00250	110	0,027	15
					264.0		19	231.9						
99	Pendimethanil	HB	Dinitroanilina	$[M+H^+]^+$	282.1	120	43	206.0	0,99	9	0,00025	86	0,006	4
					282.1		20	222.1						
100	Permethrin	IN	Piretroide	$[M+NH_4^+]^+$	408.1	83	50	153.1	0,99	13	0,00250	79	0,004	3
					408.1		20	183.1						
101	Phenothrin	IN	Piretroide	$[M+H^+]^+$	351.2	89	15	183.2	0,97	20	0,00150	37	0,005	8
					351.2		24	249.0						
102	Phorate	IN	Organotiofosfato	$[M+H^+]^+$	261.1	108	38	108.9	0,97	17	0,00500	89	0,010	6
					261.1		20	137.0						
103	Phorate sulfone	IN metabolita	Organotiofosfato	$[M+H^+]^+$	293.1	83	13	74.2	0,99	15	0,00250	100	0,003	2
					293.1		35	97.0						
104	Phorate sulfoxide	IN metabolita	Organotiofosfato	$[M+H^+]^+$	277.0	91	33	97.0	0,99	20	0,00100	88	0,002	1
					277.0		20	143.0						
105	Phosalone	IN,AC	Organotiofosfato	$[M+H^+]^+$	368.0	100	58	75.0	0,98	8	0,00250	93	0,004	3
106	Phosmet	IN	Organotiofosfato	$[M+H^+]^+$	318.2	68	34	133.0	0,98	17	0,00025	89	0,005	4
					318.2		17	160.0						
107	Phosphamidon	IN,AC	Oragnofosforato	$[M+H^+]^+$	300.1	178	25	127.1	0,99	10	0,00025	86	0,007	5
					300.1		14	174.1						
108	Pirimiphos ethyl	IN	Organotiofosfato	$[M+H^+]^+$	334.1	98	25	182.1	0,99	17	0,00025	64	0,013	13
					334.1		23	198.2						
109	Pirimiphos methyl	IN	Organotiofosfato	$[M+H^+]^+$	306.1	100	32	108.1	0,99	17	0,00025	74	0,001	1
					306.1		22	164.1						
110	Procymidone	FU	Dicarbossiamide	$[M+H^+]^+$	284.0	136	22	88.2	0,99	16	0,00500	105	0,011	6
					284.0		16	256.0						
111	Profenophos	IN	Organotiofosfato	$[M+H^+]^+$	374.8	121	19	304.9	0,99	14	0,00025	63	0,022	20
					374.8		13	346.9						

Segue Tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
112	Prometryn	HB	Triazinico	$[M+H^+]^+$	242.0	90	27	138.0	1,00	17	0,00025	54	0,002	2
					388.0		25	163.0						
113	Propyzamide	HB	Amide	[M+H+]+	256.1	113	36	145.0	0,98	18	0,00100	70	0,003	3
					256.1		24	172.9						
114	Pyraclostrobin	FU,PG	Strobilurina-Carbammato Clorurato	[M+H+]+	388.0	98	31	149.0	0,99	18	0,00050	88	0,003	2
					388.0		25	163.0						
115	Pyrazophos	FU	Organofosforato	$[M+H^+]^+$	374.1	147	33	194.0	0,99	20	0,00025	80	0,003	3
					374.1		22	222.0						
116	Pyrimethanil	FU	Anilopiridina	$[M+H^+]^+$	200.1	147	25	107.1	0,98	16	0,00100	57	0,008	8
					200.1		23	183.1						
117	Pyriproxifen	IN	Fenilureico	$[M]^+$	322.1	_	21	96.2	0,99	17	0,00025	42	0,011	15
					322.1		29	185.1						
118	Quinalfos	IN	Organotiofosfato	$[M+H^+]^+$	299.0	66	22	147.0	0,99	11	0,00050	71	0,002	2
					299.0		21	163.0						
119	Quinoxyfen	FU	Organoclorurato	$[M+H^+]^+$	308.0	213	42	162.0	0,99	17	0,00050	25	0,001	3
					308.0		32	197.1						
120	Quizalofop ethyl	HB	ArilossiFenossiPropionico	$[M+H^+]^+$	373.1	153	21	271.1	0,99	8	0,00250	70	0,002	1
					373.1		18	299.1						
121	Resmethrin	IN	Piretroide	$[M+H^+]^+$	339.2	88	41	128.2	0,98	1	0,00150	36	0,009	15
					339.2		13	171.1						
122	Rotenone	IN	Rotenoide	$[M+H^+]^+$	395.1	166	23	192.1	0,99	16	0,00025	84	0,003	2
					395.1		22	213.1						
123	Simazine	HB	Triazinico	$[M+H^+]^+$	202.0	80	16	124.0	0,98	11	0,00500	70	0,0005	4
					202.0		18	132.0						
124	Spiridiclofen	AC,IN	Acido tetronico	$[M+H^+]^+$	411.1	102	14	71.2	0,94	16	0,01000	110	0,023	12
					411.1		12	313.1						
125	Spiromesifen	AC,IN	Acido tetronico	$[M+H^+]^+$	371.1	161	35	127.2	0,99	14	0,00500	76	0,001	1
					371.1		6	330.2						
126	Spiroxamine	FU	Spirochetalamina	$[M+H^+]^+$	298.2	133	28	100.2	0,99	11	0,00025	50	0,005	6
					298.2		18	144.2						
127	Tebuconazole	FU	Conazolo	$[M+H^+]^+$	308.1	96	33	70.1	0,98	10	0,00100	72	0,009	7
					308.1		41	125.0						
128	Terbufos	IN	Organotiofosfato	$[M]^+$	288.1	189	39	65.2	0,99	12	0,01000	105	0,012	7
					288.1		31	91.2						
129	Terbufos sulfone	IN metabolita	Organotiofosfato	$[M+H^+]^+$	321.1	79	38	97.1	0,99	11	0,00250	83	0,002	1
					321.1		31	115.1						
130	Terbufos sulfoxide	IN metabolita	Organotiofosfato	$[M+H^+]^+$	305.1	73	28	131.0	0,99	18	0,00250	87	0,003	2
					305.1		11	187.1						
131	Terbuthyazine	HB	Triazinico	$[M+H^+]^+$	230.0	80	28	96.0	0,99	10	0,00025	59	0,010	11
					230.0		16	174.0						
132	Terbutryn	HB	Triazinico	$[M+H^+]^+$	242.1	78	20	186.1	1,00	6	0,00050	55	0,003	3
					242.1		17	200.0						
133	Tetrachlorvinphos	IN	Organofosforato	$[M+H^+]^+$	364.8	_	20	238.9	0,99	6	0,00150	80	0,002	1
134	Thiomethon	IN,AC	Organotiofosfato	$[M+H^+]^+$	247.1	57	38	63.1	0,98	8	0,00100	70	0,015	13

Segue Tabella III

N.	Principio attivo	Categoria	Classe	Ione pseudomolecolare m/z	Ione Parentale m/z	S-lens (Volts)	CE % (Volts)	Ioni frammento m/z	R2	Residui %	LCL mg/kg	Recupero %	r mg/kg	RSDr %
135	Tolclofos methyl	FU	Organofosforato	$[M]^+$	301.1	100	17	125.0	0,98	15	0,00250	56	0,001	1
					301.1		29	174.9						
136	Tolyfluanid	FU,AC	Fenilsufamide	$[M+NH_4^+]^+$	364.1	106	30	238.1	0,99	16	0,00100	94	0,008	5
					364.1	72	16	318.1						
137	Triazophos	IN,AC	Organotiofosfato	$[M+H^+]^+$	314.0	_	21	162.1	0,99	10	0,00025	78	0,001	1
138	Trichlorfon	IN	Organotiofosfato	$[M+H^+]^+$	257.0	_	20	127.2	0,95	15	0,05000	138	0,010	4
139	Trifloxystrobin	FU	Strobilurina	$[M+H^+]^+$	409.0	111	43	145.0	0,99	4	0,00025	103	0,003	2
					409.0		18	186.0						
140	Vinclozolin	FU	Dicarbossiamide	$[M+H^+]^+$	286.0	134	32	119.1	0,98	17	0,00100	87	0,009	6
					286.0		22	178.1						
141	Zoxamide	FU	Benzamide	$[M+H^+]^+$	336.1	126	36	159.0	0,99	10	0,00050	93	0,001	1
					336.1		21	187.0						

Legenda: AC = acaricida, DE = dessiccante, FU = fungicida, HB = erbicida, IN = insetticida, NE = nematocida, PA = attivatore pianta, PG = regolatore crescita,, RE = repellente

giunta di residui, ma solo della soluzione di standard interno. Per ciascun campione sono state eseguite due determinazioni indipendenti, ciascuna delle quali iniettata in doppio nel sistema di analisi (5 µl).

CONTROLLO SOLVENTI (bianco solventi)

È stata effettuata una preparazione senza il campione utilizzando i solventi nelle stesse proporzioni, ed il residuo ottenuto è stato iniettato nel sistema UHPLC per la verifica dell'assenza dei residui nei solventi di estrazione

CONDIZIONI STRUMENTALI

Ottimizzazione della ionizzazione in spettrometria di massa

L'ottimizzazione dei parametri di ionizzazione per ciascun residuo è stata condotta con l'infusione, ad una velocità di 5 µl/min, di ciascuna soluzione di tuning a concentrazione di 1 µg/ml in metanolo. È stata selezionata la polarità di ionizzazione e sono state determinate la S-lens di focalizzazione e l'energia di collisione ottimale di ogni singolo principio attivo effettuando uno studio di frammentazione in MS/MS. Le condizioni operative sono descritte nella Tabella II. I risultati ottenuti sono riportati nella Tabella III.

Condizioni cromatografiche per l'analisi in UHPLC/MS-MS

La separazione degli analiti è stata effettuata mediante una colonna cromatografica in fase inversa. La fase mobile utilizzata era costituita da un gradiente binario, sviluppato secondo quanto riportato nella Tabella IV e costituito da:

A. Tampone di ammonio formiato 2 mM allo 0,05% di acido formico

B. Metanolo.

RISULTATI e DISCUSSIONE

La validazione del metodo e l'analisi dei campioni di olio extravergine di oliva sono stati condotti rispettando le linee guida per il controllo qualità e le procedure di validazione per l'analisi dei prodotti fitosanitari in alimenti e mangimi riportate nel protocollo SANCO/12571/2013 [1].

La quantificazione è stata eseguita utilizzando le *curve in matrice*.

Per quanto riguarda la *linearità*, una curva di calibrazione è ritenuta idonea quando i residui risultano essere inferiori o uguali al 20%. Tutti i principi analizzati hanno mostrato valori di $R^2 \geq 0,93$ e scarti dei residui ≤ 20%. Sempre lo stesso protocollo stabilisce che il valore di LCL (*sensibilità*) debba essere uguale o inferiore al valore di MRL. Tutti i principi considerati hanno mostrato un valore di LCL compreso tra 0,00025 mg/kg e 0,050 mg/kg e comunque sempre inferiore o uguale a quello stabilito nel Reg. CE 396/2015 [2] e dal Reg. UE 788/2012 [3].

Il *recupero* degli analiti è risultato essere compreso all'interno dell'intervallo 70-120% per l'83% dei principi considerati e il valore della RSDr % della ripetibilità ≤ 20% per il 99% dei pincipi. In conclusione, la maggior parte dei principi ha soddisfatto pienamente le linee guida riportate dal protocollo SANCO utilizzato.

Gli analiti che hanno mostrato un recupero al di fuori del range atteso, ma comunque con un valore di RSD_r % inferiore al 20% sono da considerarsi comunque validati, mentre negli altri casi il valore della resa dovrà essere applicato al risultato quando il principio sarà rilevato.

Per quanto riguarda l'*analisi spettrale* per la conferma della presenza dei residui è necessaria la rivelazione di almeno 2 ioni prodotto nello spettro di frammentazione ottenuto. Il discostamento tra l'intensità degli ioni presenti nel campione e quelli presenti nello standard è legato all'intensità dello ione e comunque ± 30% come tolleranza massima. In fase di ottimizzazione il 9% dei principi ha mostrato la presenza di un solo ione frammento e in caso di rilevazione positiva di uno di questi in un determinato campione, non sarebbe possibile avere la conferma spettrale come richiesto. Sarà quindi necessario in questo caso ricorrere ad un' altra analisi confermatoria. In particolare l'Amitraz non frammenta e viene acquisito in modalità SIM(+).

Uno solo dei principi, il Methoxyfenozide, viene acquisito in modalità negativa, con un solo frammento; la maggior parte dei residui ionizza in modalità positiva nelle condizioni selezionate.

E' stato considerato un valore di incertezza estesa (Ue) pari al 50% come previsto dal protocollo SANCO considerato [1]. Dopo aver controllato tutti requisiti necessari per la conferma della presenza di un determinato principio, il valore riscontrato ottenuto dal calcolo della curva in matrice è stato sottratto dal valore dell'incertezza. Il valore risultante quando superiore al valore di MRL indicava la presenza certa del principio e la non conformità ai regolamenti [2, 3]. In ogni caso per la conformità non doveva mai superare il valore della tossicità acuta stabilito dall'ADI (dose massima giornaliera) [17].

I 23 campioni di olio extravergine di oliva sono stati analizzati con il metodo validato al fine di condurre un analisi di tipo conoscitivo del mercato e per verificare quali principi fossero ritrovati più frequentemente nei prodotti presenti nel commercio. Nella Tabella V sono riportati i risultati ottenuti.

I prodotti fitosanitari ritrovati appartengono a diverse classi chimiche: Organofosforati, Organotiofosfati, Organoclorurati, Piretroidi e Regolatori di crescita.

Per quanto riguarda gli *Organofosforati* e gli *Organotiofosfati* sono stati ricercati 58 principi. In nessun campione le concentrazioni sono risultate superiori al valore di LMR. I principi identificati più frequentemente sono quelli utilizzati come insetticidi, per combattere la mosca dell'olivo (Dacus Oleae), in particolare:

Tabella IV – Gradiente UHPLC

Tempo (minuti)	% A	% B	Flusso ml/min
0.0	80	20	300
0.5	80	20	300
6.0	5	95	300
18.0	5	95	300
19.0	80	20	300
25.0	80	20	300

Deltamethrin, Dimethoate, Formothion, Omethoate, Oxyfluorfen, Phorate, Phosmet. Nel campione 23, è stata riscontrata presenza di Deltametrhin, ma con valore inferiore al limite di legge pari a 1 mg/kg per le olive. Per quanto riguarda il Dimethoate e l'Omethoate, essi sono stati riscontrati nel campione 22, in particolare il Dimethoate è stato riscontrato in quantità pari a 0,060 mg/kg, superiore al valore del limite di legge fissato pari a 0,050 mg/kg (olive). La somma di Dimethoate e Omethoate espressa in Dimethoate è risultata essere pari a 0,061 mg/kg, inferiore al limite di legge fissato a 2 mg/kg (olive). In ogni caso per quanto riguarda il Dimethoate, dopo aver sottratto il valore dell'incertezza di misura (50%) dal valore riscontrato, il campione è risultato conforme al Reg. CE 396/2005 [2]. Tracce di Formothion sono state riscontrate nella maggior parte dei campioni (in ben 19 campioni) di cui il 14 e il 21 erano biologici (BIO). Il limite di legge per questo composto è fissato a 0,02 mg/kg nelle olive e quindi a 0,1 mg/kg nell'olio. In tutti i campioni i valori erano al di sotto del limite di legge. Il Formothion non è autorizzato in Europa dal Reg. 1107/2009 [15]. Per quanto riguarda l'Oxyfluorfen, esso è stato riscontrato in 6 campioni, ma sempre al di sotto del limite di legge (1 mg/kg per le olive). Il Phorate è stato rilevato nel campione 21 tra l'altro di dichiarata origine biologica, ma in ogni caso al di sotto del limite di legge (0,01 mg/kg per le olive). Non è autorizzato dal Reg. CE 1107/2009 [15].

Il Phosmet è stato riscontrato nei campioni N. 12 e N. 15 al di sotto del limite di legge (0,01 mg/kg per le olive).

Per quanto riguarda gli *Organoclorurati* sono stati ricercati 5 principi e l'unico dosabile è risultato essere il Chlorpyrifos ethyl nei campioni 1, 6 e 12 a concentrazione al di sotto del limite di legge (0,05 mg/kg sulle olive).

Il Daminozide, appartenente alla classe dei *Regolatori di crescita* è stato rilevato nei campioni 9, 12, 15 e 17, sempre al di sotto dei valori limite di legge (0,1 mg/kg sulle olive).

Per quanto riguarda i *Piretroidi*, sono stati ricercati 9 principi e in 5 dei 23 campioni totali (7, 12, 13, 17 e 20), è stato ritrovato il principio Fenpropathrin a concentrazioni superiori ai limiti di legge (0,01 mg/kg sulle olive). L'utilizzo di questo principio non è consentito nei territori appartenenti all'Unione Europea e quindi anche in Italia. Per poterne confermare la presenza è stata condotta l'analisi qualitativa spettrale. E' stato

Tabella V - Risultati dei campioni

Campione	Principio										
	mg/kg Chlorpyrifos ethyl OCl	mg/kg Daminozide RC	mg/kg Deltamethrin OP	mg/kg Dimethoate OP	mg/kg Omethoate OP	Dimethoate and Omethoate, somma espressa come Dimethoate mg/kg OP	mg/kg Fenpropathrin P	mg/kg Formothion OP	mg/kg Oxyfluorfen OP	mg/kg Phorate OP	mg/kg Phosmet OP
1	0,010										
2											
3								0,020			
4								0,010			
5								0,033			
6	0,014							0,019			
7							0,068	0,038			
8								0,018			
9		0,010						0,021			
10								0,044	0,010		
11								0,056			
12	0,010	0,010					0,055	0,016			0,010
13							0,090	0,016			
14								0,010			
15		0,015						0,044	0,021		0,011
16								0,024	0,025		
17		0,015					0,051	0,034	0,025		
18								0,024	0,012		
19								0,067	0,022		
20							0,052	0,012			
21								0,022		0,014	
22				0,060	0,001	0,061					
23			0,033								
LCL=LOQ	0,0010	0,0025	0,010	0,0005	0,0005	0,001	0,050	0,010	0,0010	0,005	0,00025
Limite Legge olive	0,05	0,1	1	(0,01)	(0,01)	2	0,01	0,02	1	(0,01)	(0,01)
Limite Legge olio	0,25	0,5	5	(0,05)	(0,05)	10	0,05	0,1	5	(0,05)	(0,05)

Legenda: OCl = Organo clorurati, RC = Regolatori di crescita, OP = Organo fosforati, P = Piretroidi

analizzato il rapporto tra gli ioni frammento con valore m/z pari a 125.00 (100%) e il valore m/z 350.00 (5%) ed esso rispettava le linee guida del protocollo SANCO quando comparato con lo standard in matrice accertandone la presenza solo nei campioni 13, 17 e 20. Sottraendo il valore dell'incertezza estesa (Ue) al valore riscontrato in questi tre campioni, il limite di MRL non veniva superato e quindi questi campioni risultavano conformi.

CONCLUSIONI

In questo lavoro sperimentale di tesi, con l'utilizzo della tecnica UHPLC/MS-MS, è stato possibile validare un metodo analitico soddisfacendo a pieno i requisiti previsti dal protocollo SANCO 12571/2013 [1] e che consente il dosaggio di diverse categorie di prodotti fitosanitari. Nella prima fase, sono stati ottimizzati i parametri analitici utilizzando lo spettrometro di massa in polarità positiva e negativa, e quindi determinando le condizioni e le frammentazioni ioniche distintive di ogni principio. Il parametro più critico è risultato essere la resa di estrazione che potrebbe essere migliorata utilizzando nella prima fase di estrazione, il metanolo anziché l'acetonitrile, in modo da estrarre meglio i principi più polari. Il metodo è stato poi utilizzato per analizzare campioni di olio extravergine di oliva prelevati nel territorio nazionale, ma di diversa origine, Italiana, Europea, e miscele di Europea ed Extra Europea, tra cui alcuni biologici, in modo da effettuare un'indagine conoscitiva sui prodotti presenti nel mercato. Tutti i campioni sono risultati conformi alla legge, in quanto tutti i livelli dei principi identificati erano inferiori ai valori di MRLs [2, 3, 13, 14]

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Rapidità, efficacia, precisione e certezza nell'analisi dei tensioattivi in acque di scarico

Il D.Lgs 152/2006 stabilisce i seguenti valori come limiti di concentrazione dei tensioattivi totali in acque di scarico: 4 mg/l per l'immissione in impianti pubblici di trattamento delle acque reflue, 2 mg/l per l'immissione in acque superficiali e 0.5 mg/l per il riutilizzo delle stesse (compreso l'avvio alla potabilizzazione).

A tal fine, per avere un computo corretto del "carico tensioattivo immesso", chi vuole (o deve) analizzare i tensioattivi dovrebbe quantificare e cumulare i contributi dei vari potenziali componenti: tensioattivi anionici, non ionici, cationici ed anfoteri.

I metodi ufficialmente riconosciuti gestiscono solo 2 delle 4 categorie (anionici e non ionici), richiedono lunghi tempi di esecuzione e sono piuttosto complicati da eseguire. Sono inoltre pericolosi a causa dei solventi e dei reattivi utilizzati.

Poiché sono basati su reazioni colorimetriche aspecifiche detti metodi sono inoltre interferiti, spesso costruttivamente (quindi con esito maggiorato), da un numero molto elevato di sostanze che rispondono alla reazione ma che non è detto che siano effettivamente tensioattivi.

I metodi in cuvetta estendono le possibilità analitiche anche alla categoria dei tensioattivi cationici (continuando a lasciare scoperta quella degli anfoteri), risultano decisamente più veloci da realizzare dei metodi tradizionali ma, basandosi sugli stessi principi dei suddetti (determinazione all'UV), ne subiscono dunque le stesse eventuali interferenze.

L'utilizzo della Cromatografia Liquida (HPLC) che può separare, identificare e quantificare ciascuna tipologia di tensioattivo eventualmente presente in miscela/soluzione, si propone come la via più affidabile per analizzare i tensioattivi presenti in soluzioni acquose. Il Settore Detergenti e Tensioattivi di Innovhub-SSI ha da tempo messo a punto con questa tecnica una metodologia analitica solida per l'identificazione e la quantificazione dei tensioattivi in soluzioni acquose, siano esse concentrate (prodotti detergenti), siano esse molto diluite (acque di scarico o di processo).

La metodologia risulta utile per ogni tipo di esigenza analitica in questo campo e permette di avere riscontri cromatografici sulla reale presenza di tensioattivi mediante curve di riferimento definite utilizzando standard analitici e commerciali dei più noti e diffusi tensioattivi di ciascuna specie.

La procedura per l'analisi delle acque prevede eventualmente fasi preliminari di concentrazione e purificazione automatizzate atte a costruire la miglior e più adatta aliquota di campione da sottoporre ad analisi cromatografica.

Un'analisi di questo tipo garantisce la certezza analitica della presenza/assenza di tensioattivi nei campioni analizzati e, in caso di presenza, essi sono speciati e quantificati singolarmente e non come prodotti di reazione colorimetriche (MBAS-anionici) o complessanti (BIAS-non ionici).

Ricordando che:

- i tensioattivi sono inseriti con funzione emulsionante in prodotti utilizzati da moltissime industrie come intermedi di produzione/lavorazione e che quindi non è affatto raro che possano essere presenti negli scarichi acquosi di aziende che non ne fanno uso specifico e diretto;
- moltissimi altri ingredienti comunemente in uso possono interferire con le metodologie tradizionali tanto da causare quantificazioni dei sovrastimate dei tensioattivi pur non essendo tali;
- la normativa sugli scarichi, nonché gli accordi commerciali di conferimento di acque di scarico a consorzi o società che si occupano di smaltimento sono argomento trasversale a tutto il panorama industriale italiano e non solo;

si ritiene utile sottolineare come la messa a disposizione da parte del Settore Detergenti e Tensioattivi di Innovhub-SSI di tale servizio analitico, possa essere estremamente utile su molti fronti: in caso di aperte contestazioni come supporto in fase di apertura/rinegoziazione di accordi commerciali di conferimento e in via preventiva, per un rapido, efficace e preciso monitoraggio della situazione di conformità ai parametri normativi di riferimento in questo campo specifico.

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Review

Wild rice (*Zizania sp.*): a potential source of valuable ingredients for nutraceuticals and functional foods

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Wild rice (*Zizania* sp.) is an annual cross-pollinated, emergent, aquatic grass that mainly grows naturally in the Great Lakes region of North America. The nutritional quality attributes of wild rice are superior to the conventional brown rice (*Oryza sativa* L.) in terms of higher contents of important minerals (especially phosphorous, potassium, magnesium and calcium), B-complex vitamins, vitamin E and amino acids. In addition, wild rice is reported to contain an appreciable amount of valuable compounds such as phenolics with antioxidant properties. The presence of such nutritionally bioactive substances contributes towards medicinal benefits and multiple biological activities of this specialty rice. The present review is mainly designed to focus on the detailed nutritional attributes, high-value bioactive components profile and medicinal/biological activities of wild rice, thus proposing to explore the functional food and nutraceutical potential of this food commodity.

Keywords: Wild rice lipids, α-Linolenic acid, Phytosterols, Tocols, Phenolics.

INTRODUCTION

In view of the rapidly growing human population, there is greater demand than ever to enhance production of major food crops such as rice and wheat, in order to overcome the issue of food security. The International Rice Research Institute predicted recently that 800 million tons of rice would be required by 2025 to meet the world's domestic needs [1]. In fact, the conventional rice (*Oryza sativa* L.) is the staple food for two-thirds of the world's population [2]. Besides the conventional huge scale rice consumption, there is now a revival of interest in the use of wild rice (*Zizania* sp.) as a specialty food in some parts of the world. Indeed, the utilization of wild rice is gaining popularity among consumers, and it is now commonly available in the supermarkets, restaurants and gourmet cuisine in North America. Moreover, wild rice is also used as an ingredient in a variety of foods such as soups, meat dishes, stuffing, breakfast cereals, pancakes, muffins and cookies, among others [3 - 6].
Wild rice, also known as Canadian rice, Indian rice, and water oats, belongs to the genus *Zizania*, and it is grown as an aquatic cereal grain. Four species of wild rice have been characterized: *Zizania palustris* L., *Zizania aquatica* L., *Zizania texana* H., and *Zizania latifolia* G. The first three species are native to North America while the fourth to Asia. The species *Zizania palustris* L. and *Zizania aquatica* L. are annuals whereas the others are perennials [3-5]. Wild rice species such as *Zizania palustris* L. and *Zizania aquatica* L. predominantly grow naturally in lakes, rivers, and streams in the Great Lakes region of North America (Mid-West region of United States and the Northern part of the Canadian prairies) [3, 7, 8].
Wild rice has been traditionally the most important food consumed by Native

Americans in the Great Lakes region of North America. Due to its high nutritional value and taste, wild rice gained increasing popularity during the late 20th century. As a result, the commercial cultivation started to fulfill the increased demand in the United States and Canada. In the U.S., the major wild rice producing areas include California and Minnesota, where it is mainly cultivated in paddy fields. In Canada, wild rice is usually harvested from natural bodies of water; the largest producer is Saskatchewan. Interestingly, wild rice is also produced in China, Hungary and Australia. The grain of cultivated wild rice is somewhat similar to the grain of conventional (white) rice (*Oryza sativa* L.), although it is longer, and the final color, after processing, is between black and brown. The wild rice kernel has a long and narrow cylindrical shape, with lengths from 7.5 to 18.0 mm and widths from 1.5 to 4.0 mm, and it is used dehulled usually but non-polished [7]. Like other cereals, wild rice grain contains about 74% starch and 14% proteins as the main constituents. In addition, it also contains dietary fiber (6.8%), lipids (1.7%), and ash (1.8%) [7]. The relatively high level of ash suggests that wild rice grain may serve as a good source of minerals such as potassium and phosphorus [3, 9].

Studies report that wild rice is high in minerals, vitamins, protein, starch, and dietary fiber but it is low in fat. Wild rice is gluten-free [10] and is safe for human consumption. Most importantly, wild rice is investigated to be a good source of antioxidant phytochemicals (especially ferulic, *p*-coumaric and vanilic acids), essential fatty acids (linoleic acid and α-linolenic acid) and nutraceuticals such as γ-oryzanol [6, 11].

Although wild rice is valued as a potential source of valuable phytochemicals and essential fatty acids, no comprehensive review on the detailed nutritional and phytochemicals profile and biological activities of wild rice was reported yet. Thus, the main objective of this review is to focus comprehensively on the nutritional attributes and profile of highly valuable bioactive compounds/antioxidants as well medicinal aspects of wild rice, proposing to explain further the functional dietary and nutraceutical potential of this food commodity.

BOTANICAL DESCRIPTION AND DISTRIBUTION

Zizania species are generally large seeded aquatic grass, also referred to as an anchored emergent, macrophyte, with a hollow cylindrical stem and long, narrow, blade-like leaves resembling those of wheat, oats, barley and giant cut grass [12]. These species can grow up to 2 meters high in wet lands. Inflorescence is paniculate, large terminal, and fruits are 10-20 mm long. The widely spread species of wild rice *Z. palustris* grows naturally in the Great Lakes region of United States and Canada, in shallow lakes and rivers. This plant is tall, annual, slender and has a few flowers. The average seed length is 16.7 mm.

Table I - Geographical distribution of different wild rice species

Species	Region	References
Z. palustris	North America, Southern Canada	[6], [11]
Z. aquatic	North America, Southern Canada	[6], [11], [42], [55]
Z. texana	North America (Texas)	[6], [48]
Z. latifolia	Southern Asia (China, Japan, Manchuria, New Zealand)	[6], [56], [57]

Native Americans have consumed this species as a staple food since prehistoric times. Another species, *Z. aquatica* is grown along St. Lawrence River, in the eastern and southern region of the United States. The plant is annual, tall and has average seed length of 14.3 mm. The *Z. texana* species is perennial, with small seeds, and grows naturally in St. Marcos River in Texas. This species is decumbent with many long stems, short panicles with an average seed length of 6 mm. Another species, namely *Z. latifolia* is widely grown in Southern Asian regions such as China. The plant is tall and has medium panicles. The average seed length is 7 mm. Table I shows the geographical distribution of different wild rice species.

VALUABLE NUTRIENTS AND MINERALS

Wild rice grain is considered a highly nutritious food due to the presence of a wide array of valuable nutrients required for bodily normal functions. The wild rice grain is superior in nutritive value to the brown rice (*Oryza sativa*) grain. For example, wild rice grain has twice the protein, less fat and more fiber compared with the brown (conventional) rice grain [5, 9, 13, 14]. Recent dietary guidelines recommend that about 50% of the grain products consumed should be fiber-rich, whole grain. In this regard, whole, wild rice grain can be considered as a healthy source of dietary fiber. Increased intake of dietary fiber is linked with the reduced risk of chronic diseases [13, 14].

Wild rice from Chinese and North American regions has also been found to be a very good source of different amino acids especially, glutamic acid, aspartic acid, arginine, leucine, alanine and phenyl alanine (Table II). The data in Table II shows that wild rice is an impressive source of essential minerals, especially phosphorous, potassium, magnesium and calcium. The wild rice also contains considerable levels of important vitamins such as thiamine, riboflavin and vitamin E as shown in Table II. It can be seen that the Chinese wild rice samples are mostly higher in their contents of amino acids and vitamins than the North American cultivars [5]. Vitamins and minerals are required for the maintenance of healthy life; in this regard, these components are present at higher concentrations in wild rice than white rice, supporting the nutraceutical potential of this non-traditional rice [15].

Wild rice has a granular starch streak which is quite

Table II - Nutritional components of wild rice in comparison with regular (white) rice [5]

Nutritional components	Wild rice (origin from Chinese)	Wild rice (origin from North American)	White rice
General nutrition composition (g/100g)			
Moisture	9.34 - 9.64	9.12 - 9.24	11.52
Protein	12.00 - 15.15	13.03 - 13.24	7.60
Fat	1.06 - 1.23	0.72 - 0.94	0.41
Ash	1.11 - 1.45	1.31 - 1.47	0.31
Crude Fiber	1.24 - 1.93	1.15 - 1.24	0.10
Total carbohydrate	71.16 - 75.04	74.25 - 74.29	80.15
Amino acid composition (g/100g)			
Alanine	0.62 - 0.81	0.67 - 0.69	0.30
Arginine	0.99 - 1.34	1.01 - 1.03	0.61
Aspartic acid	1.04 - 1.43	1.18 - 1.22	0.63
Cysteine	0.34 - 0.39	0.32 - 0.40	0.21
Glutamic acid	2.21 - 2.79	2.32 - 2.40	1.37
Glycine	0.57 - 0.69	0.56 - 0.58	0.30
Histidine	0.36 - 0.43	0.42 - 0.42	0.23
Isoleucine	0.44 - 0.58	0.52 - 0.53	0.28
Leucine	0.85 - 1.11	0.89 - 0.90	0.62
Lysine	0.55 - 0.73	0.59 - 0.60	0.29
Methionine	0.28 - 0.29	0.32 - 0.35	0.14
Phenylalanine	0.57 - 0.78	0.66 - 0.68	0.40
Proline	0.33 - 0.44	0.34 - 0.35	0.23
Serine	0.60 - 0.77	0.64 - 0.65	0.35
Threonine	0.40 - 0.51	0.43 - 0.43	0.23
Tryptophan	0.16 - 0.25	0.21 - 0.21	0.23
Tyrosine	0.39 - 0.53	0.43 - 0.44	0.45
Valine	0.62 - 0.83	0.68 - 0.68	0.69
Minerals (mg/100g)			
Calcium	23.26 - 24.22	21.96 - 22.81	19.25
Chromium	0.09 - 0.14	0.11 - 0.14	0.03
Cobalt	0.04 - 0.11	0.05 - 0.05	0.02
Copper	0.10 - 0.35	0.34 - 0.41	0.10
Iron	2.25 - 3.17	1.53 - 1.60	1.02
Lithium	0.02 - 0.04	0.03 - 0.04	0.01
Magnesium	106.41 - 120.91	110.90 - 119.14	47.44
Manganese	0.99 - 1.45	0.93 - 0.95	0.61
Nickel	0.02 - 0.03	0.02 - 0.03	0.03
Phosphorus	236.61 - 316.63	295.46 - 384.73	95.95
Potassium	145.59 - 232.91	237.48 - 244.91	65.18
Sodium	1.34 - 3.74	5.75 - 5.86	2.38
Zinc	1.25 - 1.98	2.51 - 2.83	0.83
Vitamins (mg/100g)			
Thiamin	0.52 - 0.61	0.36 - 0.50	0.12
Riboflavin	0.07 - 0.14	0.20 - 0.20	0.05
Vitamin E	0.20 - 0.48	0.20 - 0.20	0.10

small (2 - 8 μm) and polygonal in shape. The amylose content of this starch ranged from 21.7 to 23.8%. The wild rice starch granules were shown to have an A type X-ray diffraction pattern similar to other cereals.

Table III - Lipids content of wild rice from different regions

Region	Lipids Content(%)	References
Canada	0.74 - 1.10	[6]
North America	0.72 - 0.94	[5]
China	0.94 - 1.23	[5]
India	0.5 - 0.8	[17]
Nigeria	0.67	[14]

The wild rice swelled more at elevated temperatures indicating that wild rice starch has weaker bonding forces within the granules. Hoover et al. [7] reported that wild rice starch is hydrolyzed faster and largely by acid than standard/brown rice starch, suggesting notable chemical structural differences between the two types. In another study [16], it has been shown that white rice and processed wheat starch replaced with wild rice starch had a beneficial effect on glucose metabolism and insulin resistance in rats fed with a high-fat/cholesterol diet.

LIPID CONTENT AND FATTY ACIDS

The lipid content of wild rice from different regions is displayed in Table III. Lipids are one of the major constituents of foods; they include triacyglycerols, diacyglycerol, monoacyglycerols, free fatty acids, phospholipids, sterols, and carotenoids [14]. The lipid content of wild rice is generally described to be low when compared to other cereal grains. Although the fat content of wild rice is quite low, approximately 1% by hexane extraction it contributes significantly to the nutritive spectrum of wild rice due to presence of different bioactive compounds with medicinal value [13]. In early literature, the wild rice lipid content was generally described to be between 0.5 and 0.8% [17, 18]. The average percentage of lipids content from the wild rice is recorded to be 0.67% [18]. Recently, Przybylski et al. [6] reported a slightly higher lipid content for the commercial wild rice samples analyzed from the United States and Canada, ranged between 0.7 and 1.1%. Meanwhile, a comparison between Chinese (*Z. latifolia*) and North American (*Z. aquatica*) wild rice showed a rather small difference of lipid content for these wild rice samples of different species and origin (0.94 - 1.23% vs. 0.72 - 0.94%, respectively) [5].

The physicochemical characteristics, such as texture, melting point, mouth feel, oxidation state and appearance, as well as edible and oleo-chemical applications of plant lipids are greatly affected by the chemical composition and concentration of the constituent fatty acids [14]. The lipids composition of wild rice is notably different from the lipids of other cereals since about one third of the total fatty acids in wild rice is comprised of one essential fatty acid (α-linolenic acid) [3, 6, 13, 17]. The principal fatty acid in wild rice lipids is linoleic acid, with a content varying between 35-38%, followed by α-linolenic acid (20-31%). Together, linoleic and linolenic acids, (polyunsaturated

fats) comprise about two-thirds of the total wild rice fatty acids [6].
Interestingly, wild rice lipids are unique by comparison with white rice, wheat, and oats lipids in regard to their higher content of an essential fatty acid: α-linolenic acid (*ca.* 30%) [19, 20]. Wild rice lipids have an omega-6 to omega-3 ratio between 1.1 and 1.8, which is beneficial for health [6]. According to a study, around 60% of triacylglycerol composition of *Z. palustris* is made of palmitoyl dilinolein, palmitoyllinoleoyl linolenin, dilinoleoyl linolenin, trilinolein, and oleyllinoleoyl linolenin [21]. Because these polyunsaturated lipid moieties are highly susceptible to oxidation, they are probably responsible for the development of rancid odors in wild rice stored for a longer time under ill-controlled conditions.
A considerable variation has been recorded in the amounts of saturated, monounsaturated, and polyunsaturated fatty acids of wild rice from different regions compared with regular/ brown rice (Table IV). However, the amount of palmitic acid is similar in both types of rice (wild and brown), whereas the amount of stearic acid in regular brown rice is almost twice that of wild rice. Among the monounsaturated fats, oleic acid is the most prominent fatty acid in wild rice lipids. Among polyunsaturated fatty acids, linoleic and linolenic are predominant. The amount of linolenic acid in wild rice is 11 to 18 times higher than that observed in regular rice [6]. A comparison of the fatty acids composition of wild rice lipids with standard brown rice lipids is given in Table IV. Previously, Oelke [3] reported that wild rice lipids contain a higher proportion of linoleic and linolenic acids with a collective contribution around 68%, which is higher than that observed in other cereals [22]. Similarly, Anderson [13] reported from United States that linoleic and linolenic acids make up more than 65% of the total fatty acids of wild rice lipids. Aizawa et al. [23] found that Japanese wild rice (*Zizania latifolia*) is a rich source of linoleic (41%) and linolenic (22%) acids. Since both of the linoleic acid and linolenic acids are known to be essential fatty acids, the high level of these acids in wild rice contributes to the high nutritional quality of this food [13].

HIGH-VALUE BIOACTIVE PHYTOCHEMICALS

Wild rice has been the primary food and sacred food source of the Native North Americans and Canadians [10]. Due to the presence of a wide array of high-value phytochemicals, the U.S. Food and Drug Administration (FDA) recognizes wild rice as a whole grain believed to confer a number of health benefits, such as reduced risk of some chronic diseases: cardiovascular disease, type II diabetes, and certain cancers [15].

PHYTOSTEROLS

Phytosterols are active ingredients of several plants and play a positive role in reducing absorption of cholesterol and reducing the level of undesirable lipoproteins in human blood, thus potentially reducing the development of heart disease. It is widely accepted that diets rich in phytosterols can lower serum cholesterol levels. A direct relationship between phytosterol intake and lowering of serum cholesterol is demonstrated for phytosterol consumption between 500-2500 mg per day [24]. The daily intake of natural phytosterols is estimated to be only about 200 to 300 mg per day [25], hence, phytosterol-enriched food products are gaining better recognition in meeting the required demand and serve this purpose.
Generally, cereals are considered one of the most important sources of phytosterols in our diet but little information is reported about the occurrence of phytosterols in wild rice. Osamu et al. [21] confirmed the existence of free sterols, sterol esters, steryl glycosides, and acylsterylglycosides in wild rice. Different phytosterols are reported to be present in wild rice lipids in the order β-sitosterol > campesterol > stigmasterol. More details on phytosterol concentrations of wild rice are recently published by Przybylski et al. [6]. According to this work, the total sterol concentrations in seven North American wild rice samples ranged between 70 and 145 g/kg lipid.
The phytosterols content of wild rice lipids is about 3.5 times higher than that reported for cereal by-products such as rice bran, wheat bran, and wheat germ [26]. This seems to be a high concentration when compared with other cereals and pseudo-cereals. For

Table IV - Fatty acid composition (g/100g of total fatty acids) of wild rice lipids

	United States		Canada		Japan	
Fatty acids	**Wild rice**	**Standard brown rice**	**Wild rice**	**Standard brown rice**	**Wild rice**	**Standard brown rice**
Palmitic (16:0)	14.5	20.4	14.1 - 18.4	15.17 - 15.42	nf	nf
Stearic (18:0)	1.1	1.6	1.1 - 1.3	1.89 - 2.04	nf	nf
Oleic (18:1)	15.9	41.3	12.8 - 16.2	39.16 - 41.56	nf	nf
Linoleic (18:2 ω6)	37.7	34.5	35 - 37	35.15 - 37.45	41.0	nf
Linolenic (18:3 ω3)	30.0	1.0	20 - 31	1.67 - 1.74	22.0	nf
Reference	[13]		[6]		[23]	

nf: not found

example, Normen and co-workers [27] analyzed the concentrations of phytosterols and phytostanols in different food products from Sweden and the Netherlands. They only quantified the phytosterols, campesterol, β-sitosterol and stigmasterol. The concentration of the dominant phytosterols in different cereal foods was found to be 99 mg/100 g in buckwheat flour, 37 mg/100 g in corn flour, 23 mg/100 g rice in flour, 68 mg/100 g in rye flour, and 60 mg/100 g in whole wheat flour [27].

Campesterol, β-sitosterol and cycloartenol (which are actually stanols) are reported to be the dominant phytosterols in wild rice lipids. These sterols/stanols make up between 54 and 75% of the phytosterols in different wild rice sources. In addition, stigmasterol, clerosterol, 2,3-dehydrositosterol, Δ^5-avenasterol, gramisterol, Δ^7-avenasterol, 24-methylenecycloartanol, and citrostadienol were also detected in the wild rice of different origins [6].

Moreover, the amount of Δ^5-avenasterol in wild rice is noted to be higher than that of regular rice (Table V). Other minor sterols, namely, clerosterol (1.9-5.5%), Δ^7-avenasterol (1.7-5.0%), citrostadienol (1.0-5.0%), 23-dehydrositosterol (1.1-3.4%), and gramisterol (1.8-3.3%) have also been identified in the wild rice lipids. In this regard, wild rice can be explored as a good source of phytosterols with potential health benefits.

Table V - Sterol composition (% of total sterols) of wild rice in comparison with regular rice [6]

Phytosterol	Wild rice	Standard brown rice
Campesterol	14.3 - 17.1	14.2 - 16.8
Stigmasterol	3.7 - 6.5	10.1 - 10.4
Clerosterol	1.9 - 5.5	1.9 - 2.9
2,3 - Dehydrositosterol	1.1 - 3.4	1.6 - 2.4
β - Sitosterol	19.1 - 32.5	25.2 - 28.8
Δ^5 - Avenasterol	5.1 - 11.7	4.1 - 6.2
Gramisterol	1.8 - 3.2	1.7 - 2.4
Cycloartenol	4.7 - 12.2	9.0 - 11.7
Δ^7 - Avenasterol	1.7 - 5.0	3.1 - 3.7
24 - Methylenecycloartanol	3.2 - 36.2	13.0 - 14.0
Citrostadienol	1.0 - 5.0	3.1 - 4.9
Others	4.2 - 10.5	3.7 - 5.2

TOCOLS

A reasonable amount of tocopherols and tocotrienols, collectively known as tocols, has been detected in wild rice [28, 29]. The quantities of total tocopherols in the wild rice lipids was found to be higher than the standard refined rice bran oil (16-452 mg/kg) [28], deodorized rice bran oil (297 mg/kg) [30], and γ-oryzanol rice bran oil (123 mg/kg) [31]. Alpha-tocopherol is the main tocopherol component detected in the wild rice lipids, with amounts ranging from 142 to 2537 mg/kg. Small amount of β, γ, and δ-tocopherol (224, 386, and 608 mg/kg lipids, respectively); have also been detected in wild rice lipids. In the North American wild rice samples and in regular rice samples, over 80% of total tocopherols were found to be α and δ isomers. The total tocotrienols content of wild rice samples is higher than that of the rice bran (155-163 mg/kg), and brown rice powder (38-53 mg/kg) [32], and methanolic rice bran extract (220-460 mg/kg) [33]. These data support the high nutraceutical value of wild rice that could be used as a potential source for the isolation of tocols with antioxidant activity.

GAMMA ORYZANOL

γ-Oryzanol is a term used for the estimation of steryl ferulates in rice (*Oryza sativa* L.) or rice products. γ-Oryzanol is a complex mixture of ferulic acid esters with sterols and triterpenic alcohols [34]. However, steryl ferulates do not only occur in rice but also are present in other cereals, such as rye and wheat [35]. γ-Oryzanol has been reported to have a cholesterol-lowering effect in animals and humans [36, 37]. It is assumed that mainly free 4-desmethylsterols are responsible for the cholesterol-lowering effect [38]. This process requires deferuloylation by gastrointestinal esterases, liberating phytosterols and ferulic acid. Next to the phytosterols, ferulic acid itself may contribute to the health beneficial effects of steryl ferulates (γ-oryzanol). Przybilski et al. [6] analyzed γ-oryzanol concentration in commercial North American wild rice and reported quantities between 459 and 730 mg/kg of lipid compared with the brown rice (*Oryza sativa*), which contains γ-oryzanol in concentrations of 459 to 613 mg/kg lipid.

Moreover, according to another study, the total amount of γ-oryzanols in wild rice lipids was found to be higher than in regular rice bran oil (RBO) [32]. Rogers et al. [29] reported that the total amount of γ-oryzanol in three refined RBO ranged from 510 to 787 mg/kg. By comparison, North American wild rice contained 1.5 times more γ-oryzanol than Brazilian rice bran oil, which contained 290 mg/kg oil [30]. Results for North American wild rice, and previously published data [29-31], suggest that γ-oryzanol content depends on rice origin, variety, and growing conditions. γ-oryzanol is known to have antioxidant and cholesterol-lowering properties. Since wild rice is a rich source of these valuable components and thus can be explored as a potential health-promoting food containing nutraceuticals [39]. Recently, Aladedunye et al. [40] reported a significant difference in the γ-oryzanol profile between North American wild rice (*Zizania palustris*) and the regular brown rice samples (*Oryza sativa* L.). Compared to the regular brown rice, two additional isomers of cycloartenol ferulate were found in the oryzanol profile of wild rice, representing one extra isomer over the highest number previously reported in literature [41]. Meanwhile the amount of stigmasterol transferulate was up to 8 times higher in wild rice compared with regular brown rice.

PHENOLICS AND VOLATILE COMPONENTS

Phenolic compounds are recognized as some of the most important secondary metabolites with multiple biological properties such as antioxidant, antimicrobial, anti-inflammatory and anticancer. In addition to being found in several fruits and medicinal plant materials, these compounds are widely present in cereals, legumes and nuts [42]. Plant phenolics such as benzoic acid, cinnamic acid, flavonoids and tannins could be present in free, esterified or soluble forms. Wild rice is considered as an important source of phenolics [43]. Total phenolics content of wild rice, is reported to be 419-588 mg gallic acid equivalents (GAE)/kg, which is notably higher than in conventional rice (46 mg GAE/kg) [11, 43]. Generally, ferulic acid was identified as a major phenolic component in the wild rice. In addition, several other phenolics including *p*-coumaric, vanilic, syringic and *p*-hydroxybenzoic acids have been detected in the wild rice. The amounts of these antioxidant components in the wild rice were found to be higher than in the conventional/white rice. For example, the concentration of ferulic acid in the white rice, measured to be as high as 102.01 mg GAE/kg, was reported to be approximately 3.5 times lower than that of the wild rice samples. Also, *para*-coumaric acid level (35.03 mg/kg) in the wild rice was higher than in white rice (3.60 mg/kg). In this regard, the consumption of wild rice can contribute to the medical benefits of a healthy diet. The concentration of major phenolic components detected in the wild rice and conventional/ white rice are shown in Figure 1. Five flavonolignan glycosides were isolated from the aerial parts of *Zizania latifolia* [44]. The flavonolignan glycosides were: tricin-4'-*O*-(threo-β-guaiacylglyceryl) ether 7"-*O*-β-D-glucopyranose, tricin-4'-*O*-(*erythro*-β-guaiacylglyceryl) ether 7"-*O*-β-D-glucopyranose, tricin-7-*O*-β-D-glucopyranose, tricin-4'-*O*-(threo-β-guaiacylglyceryl) ether 7-*O*-β-D-glucopyranose, tricin-4'-*O*-(eryhtro-β-guaiacylglyceryl) ether 7"-*O*-β-D-glucopyranose.

Cho and Kays [45] assessed the volatile compounds in *Zizania palustris* and identified seventy-one volatile compounds by GC-MS and GC-olfactometry. 2-n-butylfuran, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, 3-ethyl-2,5-dimethylpyrazine, furfural, methylpyrazine and 2-pentylfuran were determined as the major volatile compounds.

BIOLOGICAL/MEDICINAL ACTIVITIES

Wild rice contains a wide array of bioactive compounds including essential fatty acids, phenolics, phytosterols, tocols, and others, which contribute to multiple biological activities of this wild food commodity. Some *in-vitro* and *in-vivo* studies demonstrated the antioxidant, and lipid-lowering effects of wild rice [1, 11, 42, 43]. Increased intake of antioxidant-rich foods has consistently been shown to be beneficial for cardiovascular disorders [46]. The dietary intake of wild rice can be used as an effective remedy for preventing hyperlipidemia. Wu et al. [47] examined the antioxidant activity of wild rice by measuring thiobarbituric acid reactivity in ground beef and by measuring peroxide values in lard. In this study, the methanol and ethanol extracts of the wild rice exhibited strong antioxidant properties. In addition to phenolics and tocols, phytic acid was determined to be an effective antioxidant compound in wild rice. In another study, Asmarai et al. [48] investigated the antioxidant activity of wild rice hull that was proven to be higher than the one of kernel extracts. According to these researchers, the potential antioxidant compounds in the hull extracts were found to be anisole, vanillin and syringaldehyde. Similarly, in another study, the antioxidant activities of wild rice and conventional white rice were evaluated by 2,2-diphenyl-1-picrylhydrazyl (DPPH)

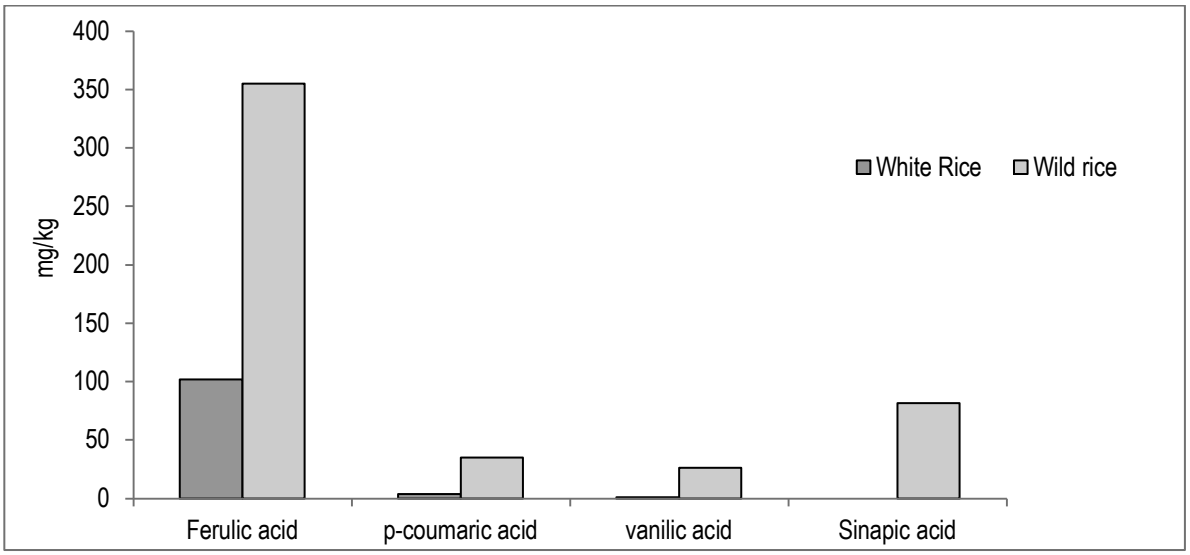

Figure 1 - Major phenolics components of white and wild rice [11]

radical scavenging and oxygen radical absorbance capacity (ORAC) assays [11, 42]. Similar to the total phenolic content, the antioxidant activity of wild rice was found to be higher than that of white rice. For example, wild rice had significantly higher ORAC values, ranging from 4454 to 11903 µmol trolox equivalents (TE)/100 g compared with white rice, which had a notably lower ORAC activity with values of 2534 µmolTE/100 g [42]. Chotimarkon et al. [33] reported on the antioxidant components and properties of wild rice and white rice cultivars. Compared with white rice, the wild rice samples contained higher amounts of phenolic compounds and exhibited stronger antioxidant properties.

In addition to the above-discussed biological activities, the wild rice has exhibited other potential activities. For instance, Zhang et al. [43] examined the effects of wild rice on serum lipid profile. They investigated the effects of replacing white rice and processed wheat starch with wild rice. In this study, wild rice suppressed the increase in serum triacylglycerol and total cholesterol. Also, wild rice intake increased superoxide dismutase activity and reduced malondialdehyde concentration in both the serum and liver. Deng et al. [49] investigated the antihypertensive effect of wild rice in spontaneously hypertensive rats and suggested that wild rice can be used in antihypertensive treatments. In addition, Surendiran et al. [50] reported that the cholesterol lowering effects of wild rice may be the main factor for the prevention of atherogenesis in LDL receptor deficient in mice. Han et al. [51] investigated the protective potential of wild rice against obesity and lipotoxicity induced by a high-fat/cholesterol diet in rats. According to their findings, wild rice has the potential of preventing obesity and liver lipotoxicity induced by a high-fat/cholesterol diet in rats.

Lee et al. [52] investigated the anti-allergic effect of the methanol extract of the wild rice species, *Z. latifolia*. The extract inhibited the release of β-hexosaminidase, which is a marker for the release of histamine by the mast cells. Lee et al. [53] investigated, in another study, the methanol extract of wild rice and found that its fractions have strong anti-allergic activities. From these reports it can be derived that wild rice may be useful for the prevention of allergic reactions. Cha [54] examined the cyctoxic effect of *Z. latifolia* rhizoma on Neuro2A cell induced by H_2O_2. According to results of this study, *Z. latifolia* rhizome inhibited the development of DNA fragmentation and apoptosis by H_2O_2. This supports that the wild rice can be used as a cytotoxic agents for pharmaceutical applications.

CONCLUSION AND FUTURE PROSPECTS

Wild rice is used as a specialty food mainly in North America. As evident from different studies, the wild rice is an excellent source of high-value components and functional bioactive substances. Wild rice has a very low lipid content (about 1%), which is quite lower than that of other cereals. More importantly, the wild rice lipids are known to be an impressive source of essential fatty acids linoleic (omega-6) and linolenic (omega-3) acids with potential health benefits. It can also be stated that among cereals, wild rice is one of the richest sources of phytosterols, especially *β*-sitosterol, campesterol and cycloartenol. As a richer source of phytosterols, the potential health benefits of wild rice towards reducing blood cholesterol and thereby preventing diseases can be understandable and needs to be clinically examined further. Moreover, it is noted that wild rice is a very good source of derivatives of tocols (tocopherols and tocotrienols), phenolics and γ-orzyanol, compounds with antioxidant properties. A high content of essential fatty acids, phytosterols, along with other high-value nutrients and antioxidant components can be linked to the nutritional and medicinal benefits of wild rice and thus supports its potential utilization for development of functional food and nutraceutical products.

Few studies reveal the antioxidant and antimicrobial activities of wild rice, however, the information about the detailed biological attributes of this valuable food are scarce. In this regard, activity-directed comprehensive studies are needed to be conducted to investigate and elucidate biological roles of different fractions of wild rice. Furthermore, there is still space for isolation, characterization and structural elucidation of different bioactive substances in wild rice, as well as elucidation of their mechanisms of biological action. Although wild rice has been utilized as a staple food by native people in different civilisations for long time, there is still a need to perform additional studies to evaluate its medicinal benefits. This review can serve as a valuable resource that explores the functional food and nutraceutical potential, as well as biological, medicinal and nutritional properties of wild rice.

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Received January 25, 2016
Accepted April 5, 2016

Analisi sensoriale

dell'Olio di Oliva vergine

Il Reg. UE 1227/2016 (modifica del Reg. CEE 2568/1991) stabilisce i parametri chimico-fisici e i metodi per il controllo di qualità dell'olio di oliva.
La valutazione organolettica (Panel test), concorre alla definizione della qualità dell'olio e alla classificazione merceologica di appartenenza.
Il Regolamento classifica l'olio di oliva vergine nelle categorie:

OLIO EXTRA VERGINE DI OLIVA
OLIO DI OLIVA VERGINE
OLIO DI OLIVA LAMPANTE

in funzione dell'intensità del fruttato, della presenza e dell'intensità di eventuali difetti.
Fornisce inoltre indicazioni sulle caratteristiche organolettiche per l'etichettatura facoltativa.
La valutazione organolettica è qualificata da un livello di affidabilità paragonabile a quello delle prove analitiche e viene eseguita da un panel di assaggiatori selezionati e addestrati, avvalendosi di tecniche statistiche per il trattamento dei dati.
Il nostro Panel è riconosciuto dal MiPAAF (Ministero delle Politiche Agricole Alimentari e Forestali) come comitato di assaggio incaricato del controllo ufficiale delle caratteristiche degli oli di oliva vergini e degli oli DOP e IGP e dal COI (Consiglio Oleicolo Internazionale).
La valutazione organolettica è accreditata da ACCREDIA (Ente Italiano di Accreditamento).
Il Panel è al servizio dell'industria, di consorzi di produzione, di enti certificatori e della grande distribuzione.

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Short note

Fatty acid composition and mineral profile of some gourds: *Lagenaria Siceraria* and *Citrullus Species* melon (egusi) seeds

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Plant sources of alpha-linolenic acid (ALA), which is converted into omega-3 fatty acids in the body, are very essential to man. Oil samples from ***Citrullus colocynthis, Citrullus vulgaris, Lagenaria siceraria*** I (African Wine Kettle gourd)***, Lagenaria siceraria*** II (Basket Ball gourd) and ***Lagenaria siceraria*** III (Bushel Giant Gourd) melon (egusi) seeds were analysed for fatty acids content using gas chromatography. Oleic acid is the main mono unsaturated fatty acid of ***Lagenaria siceraria*** and ***Citrullus sps.*** with values ranging from 8.85% ***(L.***siceraria III) to 18.46% (***C.vulgaris***) respectively. The total unsaturated fatty acid value of the gourd seeds ranges from 77.05% (***L.siceraria*** III) to 80.71% (***L.siceraria*** II). Iron content of the varieties of gourd seeds varies from 213.04 mg/kg (***Citrullus vulgaries***) to 412.19 mg/kg (*Lagenaria siceraria* II).
Keyword: *Lagenaria siceraria, Citrullus sp.,* fatty acid, micronutrients, melon, egusi

INTRODUCTION

Gourd melon (egusi) seeds were reported to be oily seeds with about 50% crude fat content [1, 2]. These melons are eaten as soup condiments in many parts of the world especially in Asia and Africa. It was reported that the total unsaturated fatty acid in *Adenopus breviflorus* benth seed is about 80.1% for the whole seeds and 77.2% for the dehulled seeds while Linoleic acid is the predominant fatty acid with values of 60.7 and 58.8% for whole and dehulled seeds oil respectively [3]. It is a general practice to fry food with oil all over the world, especially in Africa. The quality of frying depends on certain qualities of the oil, such as the fire point and smoke point. The smoke point generally refers to the temperature at which a cooking fat or oil begins to break down to glycerol and free fatty acids, and produce bluish smoke. The glycerol is then further broken down to acrolein that is a component of the smoke. The presence of the acrolein causes the smoke to be extremely irritating to the eyes and throat. Therefore, it is a key consideration when selecting a fat for frying, with the smoke point of the specific oil dictating its maximum usable temperature and therefore its possible applications. The smoke point of an oil does tend to increase as the free fatty acid content decreases and the degree of refinement increases [4].
K and Na are required by the body to maintain the osmotic balance of body fluid, the pH of the body, regulation of body muscle, nerve irritability and control of glucose absorption and enhancement of the normal retention of protein during growth. The K/Na ratio of plants seed is usually higher than 1. Hence the need to add NaCl to food, not only to improve the taste, but also to increase the Na level to enhance salt balance in the body [5]. Cucubita maxima seed powder was reported to be rich in sodium (296.90ppm), potassium (358.60ppm), magnesium (348.71 ppm), calcium (294.74 ppm)

and phosphorus (2241.45 ppm) which are essential components of body fluid, for the maintenance of the fluid balance in the body, impulse conduction, healthy muscles and nerves, bone and teeth and maintenance of normal acid-base balance of the blood respectively. It was also very low in manganese (17.93), zinc (39.85) and iron (42.70) which are trace elements essential for enzyme and glucose formation, component of insulin and wound healing and essential for the formation of red blood cells respectively [6]. This research work therefore analyses some gourds*: Lagenaria siceraria* and *Citrullus species* melon (egusi) seeds for Fatty acid, characteristic points when heated, as well as their mineral contents.

EXPERIMENTAL PROCEDURES

SAMPLE PREPARATION

Oil samples from *Citrullus colocynthis, Citrullus vulgaris, Lagenaria siceraria* I (African Wine Kettle gourd)*, Lagenaria siceraria* II (Basket Ball gourd) and *Lagenaria siceraria* III (Bushel Giant Gourd) melon (egusi) seeds were extracted using the solvent extraction method with n-hexane in soxhlet's extractor.

FATTY ACID ANALYSIS

Different fatty acids content of the melon oil samples were determined using gas chromatographic analysis, as previously described by Facchetti and Cadoppi [7]. The fatty acid methyl esters was obtained quantitatively from the oil by direct transesterification with methanolic sodium hydroxide at room temperature, followed by subsequent methylation with 14% boron trifluoride (BF_3) – methanol. Table I shows the gas chromatography specifications used for the fatty acid analysis. The reference for the fatty acids analysis is AOCS Ce 2-66 and the calculations were made according to the normalization procedure described in AOCS Ce 1f-96.

DETERMINATION OF SMOKE, FLASH AND FIRE POINTS OF OIL

Specific gravity was carried out according to the methods of AOAC [8]. The flash point and fire point of the oil sample were determined using open cup method according to NTT [9]. A metal cup containing a specified amount of the oil was heated at a rate of 5-6°C/min. The smoke point is the temperature at which smoke was just observed from the oil as the heating proceeds. The flash point is recorded as the temperature at which the material exhibits a flash when the source of ignition is swept over its surface. The sample is heated further continuing the same procedure and the fire point is recorded as the temperature at which the sample continues to burn for at least 5 seconds [9].

MINERAL ANALYSIS

The diluted ash solutions of the samples were used for the determination of the mineral content of the samples. The elements determined using atomic absorption spectrometer are: Fe, Cu, Mg, Co, Pb, Zn while Al, Na and K where determined using flame photometer. The total phosphorus in each of the sample was determined spectrophotometrically by the use of phosphovanadomolybdate [8]. Two milliliter of the diluted filtrate was pipetted and 2 ml phosphovanadomolybdate solution was added. The color developed was read at 470 nm. Standard phosphorus solution was prepared from metaphosphoric acid solution at concentration of 1000mg/l. An established regression equation through the standard was used to calculate the amount of Phosphorus in each sample. The regression equation obtained was:

$y = 36.5756x + 0.231s$

Where:

y = concentration of phosphorus (mg)

x = absorbance and coefficient of correlation

R = 0.995

Table I - Gas chromatography specifications for the determination of fatty acid profile of gourd melon (egusi) seed oils

S/N	Parameter	Description or specification
1	Perkin Elmer	XL Brown
2	Column	BPX-070-004, 30 m × 0.25 mm ID
3	Detector	FID
4	Detector Temperature	250°C
5	Injector Temperature	220°C
6	Injector	Split
7	Temperature Program	Initial Temperature 60°C (For 2 Minutes)
8	Rate of increase	10°C/min until 180°C then rate changed to 4°C/ Minute until 235
9	Carrier Gas	Helium
10	Flow Rate	15 Psi
11	Split Flow Rate	50:1
12	Total Run Time	27.7 minutes
13	Reference	AOCS Ce 2-66
14	Standard used	Kel Fir FAME 5 Standard

STATISTICAL ANALYSIS OF DATA

One-way analysis of variance (ANOVA) and least significance difference (LSD) were carried out on the replicate data generated for Fatty Acids only, using SPSS 15 packages. The results are expressed as mean ± standard deviation. Duncan was also used to determine values that are significantly different with $p \leq 0.05$ [1].

RESULT AND DISCUSSION

FATTY ACID AND OIL PROPERTIES

The fatty acid contents of the varieties of gourd melon seeds are presented in Table II. The saturated fatty acids in the oil samples of the melon seeds are myristic acid (14:0), palmitic acid (16:0), arachidic acid (20:0), behenic acid (22:0) and lignoceric acid (24:0). Most of these saturated fatty acids are lesser than 1% except for palmitic and stearic acids which range from 8.95% (*C.vulgaris*) to 12.83% (*L.siceraria* III) and 7.33% (*L.siceraria* I) to 11.09% (*C.vulgaries*) respectively. The values of the saturated fatty acids show that these varieties of melon seeds have relatively low total saturated fatty acids contents, ranging from 19.01 (*L.siceraria* I) to 22.57 (*L.siceraria* III). However, myristic and palmitic acids have been established as the most important of the dietary risk factors in cholesterol high density (CHD) [10]. High level of blood cholesterol is associated with the incidence of CHD that increase the LDL (low density lipoprotein in which 46% of the molecule is cholesterol) [10]. Stearic acid, the other main saturated fatty acid, does not have this effect because it can be converted to oleic acid which is a monounsaturated fatty acid [10]. Hence, the CDH factor in these varieties of melon seeds will only range from 9.54% (*C.vulgaries*) to 12.37% (*L.siceraria* I), which is relatively low. The mono unsaturated fatty acids in the five varieties of gourd melon seed oil samples are palmitoleic acid (16:1), oleic acid (18:1) and eicosenic acid (20:1). Oleic acid is the main mono unsaturated fatty acid of these seeds, with values ranging from 8.85% (*L.siceraria* III) to 18.46% (*C.vulgaris*) respectively. The polyunsaturated fatty acid is responsible for the greatest percentage of the fatty acid content of the melon seeds oil with linoleic acid (18:2) having values ranging from 60.54% (C.vulgaries) to 68.98% (*L.siceraria* I), while linolenic acid (18:3) has values ranging from 0.12% (C. colocynthis) to 0.17% (L.siceraria I) respectively. Therefore, the total unsaturated fatty acid value of the gourd seeds ranges from 77.05% (*L.siceraria* III) to 80.71% (*L.siceraria* II), Table II.

Total essential fatty acid (linolenic + lenoleic acid) is therefore higher and compare favorably with the total essential fatty acid in soybean (59.40%) and pigeon pea (60.40%) [11]. None of these melon seed oil samples exhibits any trans fatty acid, but only cis fatty acids which makes the melon seed oil heart friendly and suitable especially for patients with heart problems. The varieties of gourd melon seed oil sample

Table II - Fatty acid profile of some varieties of gourd seed oil samples (%)

	Sample				
Fatty acid	***C.colocynthis***	***C.vulgaris***	***L.siceraria* I**	***L.siceraria* II**	***L.siceraria* III**
Myristic acid (14:0)	$0.00^{a} \pm 0.00$	$0.02^{ab} \pm 0.03$	$0.09^{b} \pm 0.01$	$0.03^{ab} \pm 0.05$	$0.07^{ab} \pm 0.06$
Palmitic acid (16:0)	$9.58^{a} \pm 0.02$	$8.95^{a} \pm 0.17$	$11.69^{a} \pm 0.19$	$11.19^{a} \pm 0.13$	$12.83^{a} \pm 0.32$
Palmitoleic acid (16:1)	$0.15^{b} \pm 0.04$	$0.03^{a} \pm 0.04$	$0.05^{a} \pm 0.04$	$0.00^{a} \pm 0.00$	$0.02^{a} \pm 0.03$
Stearic acid (18:0)	$10.33^{c} \pm 0.17$	$11.09^{d} \pm 0.30$	$7.33^{a} \pm 0.10$	$7.33^{a} \pm 0.26$	$9.15^{b} \pm 0.02$
Oleic acid (18:1)	$15.81^{d} \pm 0.11$	$18.46^{e} \pm 0.26$	$10.59^{b} \pm 0.07$	$13.84^{c} \pm 0.26$	$8.85^{a} \pm 0.04$
Linoleic acid (18:2)	$63.12^{b} \pm 0.19$	$60.54^{a} \pm 0.54$	$68.98^{d} \pm 0.34$	$66.69^{c} \pm 0.88$	$67.89^{d} \pm 0.41$
Linolenic acid (18:3)	$0.12^{a} \pm 0.02$	$0.13^{a} \pm 0.00$	$0.17^{a} \pm 0.01$	$0.13^{a} \pm 0.10$	$0.14^{a} \pm 0.02$
Arachidic acid (20:0)	$0.45^{ab} \pm 0.01$	$0.43^{a} \pm 0.02$	$0.42^{a} \pm 0.03$	$0.46^{ab} \pm 0.02$	$0.51^{b} \pm 0.05$
Eicosenic acid (20:1)	$0.14^{a} \pm 0.00$	$0.12^{a} \pm 0.09$	$0.19^{a} \pm 0.07$	$0.15^{a} \pm 0.11$	$0.15^{a} \pm 0.02$
Behenic acid (22:0)	$0.13^{b} \pm 0.01$	$0.10^{ab} \pm 0.02$	$0.12^{b} \pm 0.02$	$0.03^{a} \pm 0.01$	$0.08^{a} \pm 0.12$
Lignocericacid (24:0)	$0.11^{a} \pm 0.00$	$0.06^{a} \pm 0.04$	$0.14^{a} \pm 0.01$	$0.08^{a} \pm 0.12$	$0.12^{a} \pm 0.08$
Unknown	$0.00^{a} \pm 0.00$	$0.06^{a} \pm 0.08$	$0.28^{a} \pm 0.06$	$0.07^{a} \pm 0.12$	$0.15^{a} \pm 0.14$
TEFA	63.24	60.67	69.15	66.82	68.03
TSFA	20.65	20.63	19.70	19.01	22.57
TMUFA	16.10	18.61	10.83	13.99	9.02
TPUFA	63.24	60.67	69.15	66.82	68.0
TUFA	79.34	79.28	79.98	80.71	77.05
TPUFA/TSFA	3.06	2.94	3.51	3.52	3.01
TUFA/TSFA	3.84	3.84	4.06	4.25	3.41

Values with different superscripts on the same row are significant at ($p \leq 0.05$). Total essential fatty acid (TEFA); Total saturated fatty acid (TSFA); Total mono unsaturated fatty acid (TMUFA); Total polyunsaturated fatty acid (TPUFA); Total unsaturated fatty acid (TUFA).

are closely related in total essential fatty acids (TEFA), total monosaturated fatty acid (TMUFA), total unsaturated fatty acid (TUFA), total polysaturated fatty acid (TPUFA) and in the unidentified fatty acid with no significant difference, $p \leq 0.05$. There are differences in their coefficient of variations showing unequal distribution in the different fatty acid composition of the varieties of melon seed oil.

The oil samples of the gourd seeds are light yellow in color with specific densities (g/dm^3): 0.92, 0.84, 0.93, 0.92 and 0.93 for *C. colocynthis, C. vulgaris, L. siceraria I, L.siceraria* II and *L. siceraria* III respectively. The smoke, flash, boiling and fire point (°C) of the seeds range from (60-150), (56-140), (110-180), and (150- 360) respectively, Table III. The high smoke and fire points make them suitable for frying and easy handling during cooking.

MINERAL CONTENT OF GOURD SEEDS

Table IV presents the mineral content of the five varieties of gourd seeds. Potassium has the highest concentration for all the seeds. High amount of potassium in food is desirable since the diet is the only source of potassium for the body [12]. The value of potassium ranges from 3,332.61 mg/kg (*Citrullus vulgaris*) to 8,550.94 mg/kg (*Lagenaria siceraria* I). This result is consistent with the report of Oshodi et al., [13] with the conclusion that potassium is the most abundant mineral in Nigerian agricultural products. Magnesium is the second most abundant element in these samples with values ranging from 3,046.50 mg/kg (*Citrullus vulgaris*) to 8,220.75 mg/kg (*Lagenaria siceraria* I). These seeds are therefore good sources of magnesium to meet magnesium needs of man and supplement for magnesium in food substances that are deficient in magnesium. These values are higher than the values of magnesium reported for benniseed (480 mg/kg), pearl millet (1,050 mg/kg) and quinoa (2,320 mg/kg) [13]. Hence, these varieties of gourd seeds adequately meet the need of magnesium in man. Sodium content of the gourd melon seeds ranges from 168.18 mg/kg (*Lagenaria siceraria* III) to 688.00mg/kg (*Lagenaria siceraria* I). These values are comparable with the sodium content of some seeds like benniseeds 316 mg/kg [13]. Calcium is an important mineral for bone and teeth formation [14], as well as for body structure and in blood clothing [15]. The values of calcium in these varieties of gourd melon seeds range from 10.61 mg/kg (*Lagenaria siceraria* III) to 413.21 mg/kg (*Lagenaria siceraria* I). These values are low and do not meet the RDA of calcium for man which is 80 mg. The values of Ca in this work for the five varieties of gourd seeds are comparable with the calcium content reported by Adeyeye et al [16].

Phosphorus content of the seeds varies from 207.50 mg/kg (*L.siceraria* I) to 521.70 mg/kg (*C.vulgaris*). This range of values is close to the phosphorus content reported by Oshodi et al., [13] for seeds like benniseed (310 mg/kg) and quinoa seed (220 mg/kg) and higher than that reported for pearl millet (99 mg/

Table III - Some characteristic point of oil extracts of the five varieties of gourd melon (egusi) seed oil samples

Characteristic points	Sample				
	C.colocynthis	***C.vulgaris***	***L.siceraria* I**	***L.siceraria* II**	***L.siceraria* III**
Specific Gravity	0.92	0.84	0.93	0.92	0.93
Smoke Point (°C)	60-150	50-100	140.00	118.00	85-150
Flash Point (°C)	60.00	56.00	120.00	140.00	90.00
Boiling Point (°C)	120.00	110.00	150.00	180.00	170.00
Fire Point (°C)	180.00	150.00	270.00	360.00	360.00
Colour	Light yellow	L. yellow	L. yellow	L. yellow	L. yellow

Table IV - Mineral content of some varieties of gourd melon (egusi) seeds (mg/kg)

Element	Sample				
	C.colocynthis	***C.vulgaris***	***L.siceraria* I**	***L.siceraria* II**	***L.siceraria* III**
Sodium	406.59	260.87	688.00	300.00	168.18
Potassium	3,474.73	3,332.61	8,550.94	6,402.44	4,390.91
Calcium	191.43	203.91	413.21	264.88	10.61
Magnesium	3,046.50	3,295.65	8,220.75	4,558.54	3,428.78
Phosphorus	439.56	521.70	207.50	487.80	378.80
Iron	281.32	213.04	311.32	412.19	380.30
Aluminium	6.59	104.35	169.81	73.17	22.73
Copper	0.00	2.17	107.55	53.66	18.18
Zinc	206.55	258.70	950.94	687.80	557.58
Lead	0.00	0.00	0.26	0.00	0.00
Manganese	0.00	0.00	0.00	0.00	0.00

kg). These values are not adequate enough to meet the Recommended Daily Allowance for phosphorus in man (80 mg/kg) if used as a soup thickener. Iron is very important for the formation of hemoglobin and the normal functioning of the central nervous system. The iron content of the varieties of gourd seeds varies from 213.04 mg/kg (*Citrullus vulgaries*) to 412.19 mg/kg (*Lagenaria siceraria* II).

The daily requirement of iron for children ranges from 10-15 mg, women and men, 18 mg and 12 mg respectively [12]. These values are met with the consumption of 4.69 mg by children, 9 mg by women and 5.63 mg by men of these gourd melon seeds. Iron contents recorded in this study for the five varieties of gourd melon (egusi) seeds are higher than those reported for breadnut (110.91-265.85) mg/kg, cashew nut (90.01-158.30) mg/kg [12].

Cupper ranges from 0.00 mg/kg (*Citrullus colocynthis*) to 107.55 mg/kg (*Lagenaria siceraria I*); Lead (0.00 - 0.26) mg/kg while manganese was not detected in the five varieties of gourd melon (egusi) seeds. The 2 mg daily requirement of copper [12] are met by consuming a maximum of 18.60 mg melon seeds as can be easily consumed by taking the melon seeds as soup thickener.

CONCLUSION

These five varieties of gourd melon seeds are rich in polyunsaturated fatty acid, with the total unsaturated fatty acid value ranging from 77.05% (*L.siceraria* III) to 80.71% (*L.siceraria* II). These melon seed oil samples are free of trans fatty acid, but all contain only cis fatty acids which makes the melon seed oil samples heart friendly and suitable especially for patients with heart problems. These seeds are therefore good sources of magnesium, sodium calcium and iron that are essential for the proper functioning of organs in human.

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Received December 30, 2015
Accepted April 8, 2016

INNOVHUB
STAZIONI SPERIMENTALI
PER L'INDUSTRIA

Innovazione e ricerca

LA STABILITÀ ALL'OSSIDAZIONE

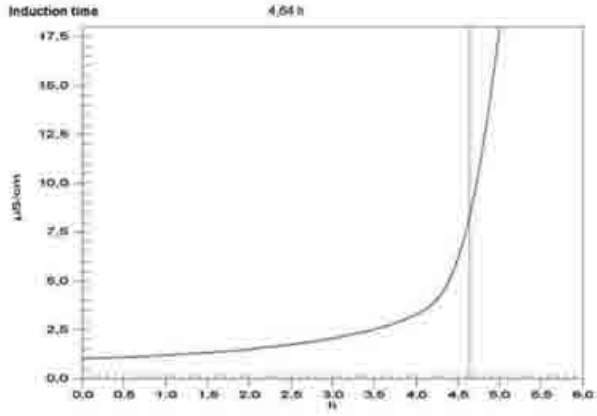
I lipidi costituiscono un ingrediente significativo delle formulazioni cosmetiche e ne determinano in particolare la conservabilità nel tempo. Le sostanze grasse infatti possono subire un fenomeno ossidativo con formazione di composti volatili la cui soglia di percezione olfattiva è estremamente bassa e che possono influire notevolmente sull'accettabilità del prodotto da parte del consumatore. Inoltre il lipide utilizzato come ingrediente di un prodotto cosmetico, subisce fasi di preparazione che possono comportare ulteriore apporto di ossigeno, nonché aggiunta di componenti che possono intervenire nel processo ossidativo con il ruolo di pro o antiossidanti. Il successivo confezionamento pone altri problemi di conservazione, legati all'ossigeno disciolto, al tipo di contenitore, alle condizioni di stoccaggio.
Per questi motivi è fondamentale valutare la stabilità all'ossidazione dei prodotti finiti oltre che delle materie prime.

Presso il **laboratorio Cosmetica** è possibile determinare la stabilità all'ossidazione di materie prime e prodotti finiti utilizzando le seguenti apparecchiature:

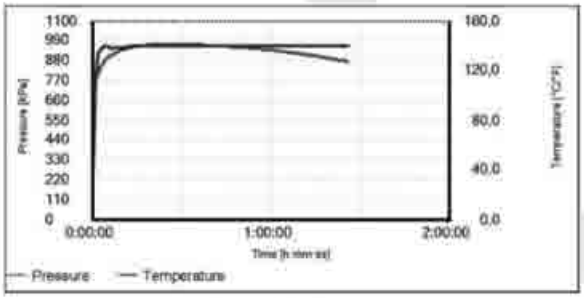
RANCIMAT

Per sostanze grasse e prodotti a base prevalentemente grassa.

Il suo impiego è previsto anche dal metodo ISO 6886.



Curva Rancimat



Curva Petroxy

PETROXY

Per sostanze grasse e prodotti finiti anche contenenti acqua o sostanze volatili che possono interferire nella determinazione con il Rancimat.

Con entrambe le tecniche si ottiene un tempo di stabilità all'ossidazione del prodotto sottoposto a test, utile per verificarne la stabilità nel tempo o valutarne le differenti formulazioni.

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Nota tecnica

Lubrificanti
Corrispondenze tra metodi analitici

(gennaio-dicembre 2016)

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Da diversi anni viene pubblicata una guida, a disposizione di chi lavora nel settore dei lubrificanti, in cui sono riportati i controlli maggiormente utilizzati per la caratterizzazione dei prodotti petroliferi e lubrificanti e i relativi metodi di analisi pubblicati da Enti Nazionali ed Internazionali (UNI, CEI, ASTM, IP, ISO, IEC, EN).

Quest'anno è stata fatta la revisione della tabella con un aggiornamento di tutti i metodi pubblicati da gennaio a dicembre 2016.

La struttura base della tabella non è stata modificata rispetto alla versione precedente: nella prima colonna si riporta il parametro analitico, cui corrispondono i numeri di norma/metodo riportati nelle colonne successive.

I riferimenti normativi sono sempre divisi in quattro classi: EN - ISO - IEC; Metodi Italiani (UNI - UNI EN - UNI EN ISO - CEI – NOM); IP; ASTM.

Tutti i metodi che durante l'anno hanno avuto revisioni o modifiche sono evidenziati con lo sfondo grigio.

La nuova versione dei metodi ASTM è stata confrontata con l'edizione precedente e nel foglio "Commento alle nuove revisioni" si riportano i risultati di tale confronto. Quando compare la dizione "equivalente" significa che c'è una perfetta rispondenza tra le metodiche; differenze non sostanziali tra i vari metodi sono riassunte nell'espressione "tecnicamente equivalenti"; per i metodi in cui è stata riscontrata anche una sola, ma significativa differenza, viene riportata l'espressione "non equivalente".

Per i metodi IP si rimanda al sito http://ein.powerweb.co.uk/cssiptmqbe.htm dove è disponibile l'elenco aggiornato dei metodi e un loro confronto con i metodi ASTM e ISO.

Preso atto della velocità di cambiamento dei metodi in ambito normativo, soprattutto dei metodi ASTM, si ricorda che la presente guida, non potendo essere aggiornata in tempo reale, ma facendo riferimento ad una valutazione temporale pari a un anno solare, ha delle lacune, insite proprio nella modalità in cui è stato concepito il lavoro di revisione. Per questo motivo alcuni metodi ASTM hanno come data di revisione il 2015, anche se l'ultima ricerca condotta a Dicembre 2015 non li citava come metodi in revisione (la ragione è da imputare ad un lasso di tempo che intercorre tra l'approvazione del metodo e la sua pubblicazione).

TABELLA LUBRIFICANTI (GENNAIO - DICEMBRE 2016)
CORRISPONDENZA TRA METODI ANALITICI

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
ACQUA IN LIQUIDI ISOLANTI (KF)	60814:1997	CEI EN 60814:1998		1533-12
ACQUA IN PRODOTTI PETROLIFERI (KF)	**12937:2000**			6304-16e1
ACQUA NEGLI ANTIFREEZES CONCENTRATI (KF)				1123-99(2015)
ACQUA PER DISTILLAZIONE				95-13e1
ACQUA NEGLI OLI ISOLANTI NELLA CARTA E NEL CARTONE IMPREGNATI OLIO	60814:1997	CEI EN 60814:1998		
ADDITIVI ANTIOSSIDANTI SPECIFICI NEGLI OLI ISOLANTI	60666:2010	CEI EN 60666:2011		
ALCALINITÀ DI RISERVA PER ANTICONGELANTI E ANTIRUGGINI				1121-11
ALTERABILITÀ DI OLI ISOLANTI	**7624:1997** 60962:1988	CEI 10-8:1997		
ANALISI DI GRASSI LUBRIFICANTI				2269-10(2015)
ASSORBIMENTO UV DI PRODOTTI PETROLIFERI				2008-12
AZOTO (CHEMILUMINESCENZA)				4629-12
AZOTO (KJELDAHL MODIFICATO)				3228-08 (2014)
BENZINA IN LUBRIFICANTI USATI (GC)				3525-04 (2016)
CALCOLO DELLA COSTANTE DI VISCOSITÀ-GRAVITA' (VGC)				2501-14
CAMPIONAMENTO DI GAS IN OLIO	60475:2011	CEI EN 60475:2012		
CARATTERISTICHE ANTIRUGGINE				665-14e1
CENERI DA PRODOTTI PETROLIFERI				482-13
CENERI NEGLI ANTICONGELANTI E ANTIRUGGINI				1119-05(2015)
CENERI SOLFATATE	**3987:2010/ Cor 1:2011**	UNI 20021:1989	163/12	874-13a
CLASSIFICAZIONE DI LIQUIDI ISOLANTI IN BASE AL PUNTO DI COMBUSTIONE E P.C. INFERIORE	61100:1992	CEI EN 61100:1997		
CLASSIFICAZIONE GENERALE DI LIQUIDI ISOLANTI	61039:2008	CEI EN61039:2009		
CLORO NEGLI OLI GREZZI				4929-16
CLORO NEGLI OLI USATI		NOM 161:2007		

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
CLORO (METODO DI DECOMPOSIZIONE AD ALTA PRESSIONE)				808-16
CLORO IONICO O IDROLIZZABILE (IN ASKAREL)	60588:1979	CEI 10-6:1997		
COLORE A S T M	**2049:1996**	UNI 20026:1989	196/97(14)	1500-12
COLORE (METODO AUTOMATICO "TRISTIMOLO")				6045-12
COLORE SAYBOLT				156-15
COLORE APHA HAZEN (per ASKAREL)	60588:1979	CEI 10-6:1997		
CONTAMINAZIONE IN DISTILLATI MEDI	*12662:2014*			
CONTAMINAZIONE DA PARTICELLE SOLIDE	**4406:1999**			
CONTENUTO DI OLIO NELLE PARAFFINE	**2908:1974**			721-15
COPPIA DI SPUNTO E ROTOLAMENTO GRASSI (A BASSA TEMPERATURA)				1478-11
CORROSIONE DI GRASSI CON LAMINA DI RAME		UNI 20035:1992		4048-10
CORROSIONE CON LAMINA DI RAME	**2160:1998**	UNI EN ISO 2160:2001	154/00(13)	130-12
DEMULSIVITÀ DI OLI				2711-11
DEMULSIVITÀ DI OLI MINERALI E SINTETICI	**6614:1994**	UNI ISO 6614:2001		1401-12e1
DENSITÀ O DENSITÀ RELATIVA DI LIQUIDI REFRIGERANTI				1122-16
DETERMINAZIONE DELLE CARATTERISTICHE DI OSSIDAZIONE DI OLI INIBITI E FLUIDI – TOST TEST Parte 1 – Oli Minerali Parte 2 – Fluidi idraulici HFC Parte 3 – Procedura anidra per fluidi idraulici sintetici Parte 4 – Oli per cambi industriali	 **4263-1:2003** **4263-2:2003** **4263-3:2015** **4263-4:2006**	 UNI EN ISO 4263-1:2005 UNI EN ISO 4263-2:2005 UNI EN ISO 4263-3:2016 UNI EN ISO 4263-4:2006		
DILAVAMENTO CON ACQUA DI GRASSI		UNI 20055:1993		1264-16
DILUIZIONE BENZINA DI OLIO USATO (DISTILLAZIONE)		UNI 20046:1992		322-97 (2016)
DISTILLAZIONE A PRESSIONE ATMOSFERICA DI PRODOTTI PETROLIFERI E LIQUIDI COMBUSTIBILI	**3405:2011**			86-16a
DISTILLAZIONE SOTTO VUOTO				1160-15
ELEMENTI DI ADDITIVAZIONE, METALLI DI USURA E CONTAMINANTI IN OLI LUBRIFICANTI USATI E OLI BASE (ICP-AES)				5185-13e1
ELEMENTI DI USURA E CONTAMINANTI IN OLI LUBRIFICANTI USATI O FLUIDI IDRAULICI USATI				6595-16
ELEMENTI DI ADDITIVAZIONE IN OLI LUBRIFICANTI (ICP-AES)				4951-14

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
ELEMENTI, Ba-Ca-S-P-Zn IN OLI LUBRIFICANTI (FLUORESCENZA RAGGI X)				4927-15
ELEMENTI, Ba-Ca-Zn-Mg IN LUBRIFICANTI NUOVI (A.A.)				4628-14
FATTORE DI DISSIPAZIONE DI LIQUIDI ISOLANTI	60247:2004	CEI EN 60247:2004		
FOSFORO IN LUBRIFICANTI ED ADDITIVI (OSSIDAZIONE)				1091-11(2016)
FOSFORO IN OLI E ADDITIVI (CHINOLINA FOSFOMOLIBDATO)	**~~4265:1986~~**	UNI 20056:1993	149/93(03)	4047-13
GAS DISCIOLTI NELL'OLIO DI TRASFORMATORI (INTERPRETAZIONE ANALISI)	60599:2015	CEI EN 60599:2000 CEI EN 60599/A1:2008		
GASOLIO IN LUBRIFICANTI USATI (GC)				3524-14
GUIDA AL CONTROLLO E TRATTAMENTO OLI MINERALI ISOLANTI IN APPARECCHIATURE ELETTRICHE	60422:2013	CEI EN 60422:2014		
INDICE DI RIFRAZIONE	**5661:1983**			1218-12(2016)
INDICE VISCOSITÀ, CALCOLO	**2909:2002**	UNI ISO 2909:2001	226/04(14)	2270-10(2016)
INSOLFONABILE, RESIDUO				483-04 (2014)
INSOLUBILI IN OLI USATI				893-14
INSOLUBILI IN PENTANO				4055-04 (2013)
INVECCHIAMENTO E VALUTAZIONE CONRADSON	**6617:1994**	UNI 20007:1989		
MASSA VOLUMICA (DENSIMETRO DIGITALE)	**12185:1996/ Cor 1:2001**		365/97(04)	4052-15
MASSA VOLUMICA	**3675:1998**	UNI EN ISO 3675:2002	160/99	1298-12b
MISCIBILITÀ OLI 2 TEMPI				4682-13
MONITORAGGIO DI LUBRIFICANTI IN ESERCIZIO CON TECNICA FT-IR				ASTM E2412-10
MONITORAGGIO DI OLI MINERALI PER TURBINE A VAPORE E A GAS				4378-13
NAFTENI IN FRAZIONI SATURE (REFRACTIVITY INTERCEPT)				~~2159-93~~
NUMERO ACIDITÀ E BASICITÀ (TITOLAZIONE CON INDICATORE)	**6618:1997/ Cor 1:1999**		139/98(04)	974-14e2
NUMERO ACIDITÀ,VALORE DI NEUTRALIZZAZIONE (TITOLAZIONE CON INDICATORE)			1/94(04)	
NUMERO DI ACIDITÀ (TITOLAZIONE POTENZIOMETRICA)	**6619 :1988**	UNI 20025:1989 UNI EN 12634:2001	177/13	664-11ae1

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
NUMERO DI ACIDITÀ SEMI-MICRO (TITOLAZIONE CON INDICATORE)	**7537:1997**			3339-12
NUMERO DI BASICITÀ (TITOLAZIONE POTENZIOMETRICA CON ACIDO CLORIDRICO)				4739-11
NUMERO DI BASICITÀ (TITOLAZIONE POTENZIOMETRICA CON ACIDO PERCLORICO)	**3771:2011**	UNI 20002:1989	276/12	2896-15
NUMERO DI NEUTRALIZZAZIONE DI OLI ISOLANTI	62021-1:2003 *62021-1:2007*	CEI EN 62021-1:2005 CEI EN 62021-2:2007		
NUMERO DI PRECIPITAZIONE PER LUBRIFICANTI				91-02 (2012)e1
NUMERO DI SAPONIFICAZIONE DI PRODOTTI PETROLIFERI	**6293-1:1996** **6293-2:1998**	UNI ISO 6293-1-2:2001	136S1/98(06) 136S2/99(06)	94-07 (2012)e1
OSSIDAZIONE DI GRASSI (BOMBA)			142/85(15)	942-15
OSSIDAZIONE DI OLI INIBITI				943-04a (2010)e1
OSSIDAZIONE DI OLI LUBRIFICANTI			48/12	
OSSIDAZIONE DI OLI LUBRIFICANTI "EP"				2893-04 (2014)e1
PCBs IN OLI MINERALI USATI (GC) -QUANTIFICAZIONE	*12766-2:2001*	UNI EN 12766-2:2004		
PCBs IN OLI MINERALI USATI (GC+ECD)	*12766-1:2000*	UNI EN 12766-1:2001		
PCT E PCBT IN OLI MINERALI USATI (GC+ECD)	*12766-3:2004*	UNI EN 12766-3:2005		
PENETRAZIONE DI GRASSI CON CONO	**2137:2007**	NOM 38:2002	50/12	217-16
PENETRAZIONE DI GRASSI CON CONO A SCALA 1/4 E 1/2		UNI 20033:1992		1403-10
PENETRAZIONE DI PARAFFINE CON AGO		UNI 20004:1989		1321-16a
PENETRAZIONE DI PETROLATI CON CONO	**2137:2007**		179/79(04)	937-07 (2012)
PENTACLOROBIFENILI E OMOLOGHI MAGGIORMENTE CLORURATI (in ASKAREL)	60588:1979	CEI 10-6:1997		
PERDITA PER EVAPORAZIONE (NOACK)				5800-15a
PERDITA PER EVAPORAZIONE DI OLI E GRASSI				972-16
PERSISTENZA DELLA FIAMMELLA IN FLUIDI RESISTENTI AL FUOCO	**14935:1998**	UNI EN ISO 14935:2000		
pH DI ANTICONGELANTI E ANTIRUGGINI				1287-11
POLARI, AROMATICI E SATURI IN OLI PLASTIFICANTI ED ESTENSORI (METODO CROMATOGRAFICO)				2007-11(2016)
POLICLOROBIFENILI IN OLI MINERALI ISOLANTI (GC impaccata)				4059-00 (2010)
POLICLOROBIFENILI IN OLI MINERALI ISOLANTI (GC capillare)	61619:1997	CEI EN 61619:1998		

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
POLINUCLEARI AROMATICI IN OLI USATI		UNI 20030:1992	346/92(04)	
PRODOTTI PETROLIFERI, TABELLE DI CONVERSIONE				1250-08 (2013)e1
PROPRIETÀ "EP" DI OLI (MACCHINA 4 SFERE)		UNI 20029:1992	239/14	2783-03 (2014)
PROPRIETÀ "EP" DI GRASSI (MACCHINA 4 SFERE)				2596-15
PUNTO DI ANILINA				611-12(2016)
PUNTO DI CONGELAMENTO DI FLUIDI REFRIGERANTI PER MOTORI				1177-16
PUNTO DI EBOLLIZIONE DI FLUIDI REFRIGERANTI PER MOTORI				1120-16
PUNTO DI FUSIONE DI PARAFFINE	**~~3841:1977~~ 6244:1982**	UNI ISO 3841:2001		87-09 (2014)
PUNTO DI GOCCIOLAMENTO DI CERE E PETROLATI	**6244:1982**	UNI 20034:1992	133/79(01)	127-08(2015)
PUNTO DI GOCCIOLAMENTO DI GRASSI	**2176:1995/ Cor 1:2001**		396/14 AUTOMATICO	566-16
PUNTO DI GOCCIOLAMENTO DI GRASSI CON PIÙ ALTO RANGE DI TEMPERATURA				2265-15
PUNTO DI INFIAMMABILITÀ IN VASO APERTO CLEVELAND	**2592:2000**		36/02	92-16b
PUNTO DI INFIAMMABILITÀ IN VASO CHIUSO (PENSKY MARTENS)	**2719:2016**		34/03	93-16a
PUNTO DI INFIAMMABILITÀ TAG (aperto)				1310-14
PUNTO DI INFIAMMABILITÀ TAG (chiuso)				56-16a
PUNTO DI INTORBIDAMENTO (RAFFREDDAMENTO LINEARE)	**3015:1992**			2500-16a
PUNTO DI SCORRIMENTO	**3016:1994**	UNI 20065:1997	15/95(04)	97-16
PUNTO DI SCORRIMENTO AUTOMATIZZATO				6892-03 (2014)
PUNTO DI SOLIDIFICAZIONE DI PARAFFINE E PETROLATI	**2207:1980**	UNI 20005:1989	76/70(04)	938-12
RESIDUO CARBONIOSO CONRADSON	**6615:1993**			189-06 (2014)
RESIDUO CARBONIOSO RAMSBOTTOM	**4262:1993**	UNI 20042:1992		524-15
RESIDUO CARBONIOSO, METODO MICRO	**10370:2014**	UNI EN ISO 10370:2015		4530-15
RIGIDITÀ DIELETTRICA DI OLI ISOLANTI	60156:1995			
RILASCIO ARIA DI OLI BASE IDROCARBURICI	**9120:1997**	NOM 121:2002		3427-15
RUGGINE, PROVA DINAMICA PER GRASSI (EMCOR)		UNI 20036:1992		

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
SCHIUMEGGIAMENTO DI ANTICONGELANTI				1881-97 (2009)
SCHIUMEGGIAMENTO DI OLI	**6247:1998/ Cor 1:1999**	UNI 20023:1989	146/10	892-13e1
SEDIMENTI IN TRACCE NEGLI OLI LUBRIFICANTI				2273-08 (2016)
SEPARAZIONE DI OLIO DA GRASSO LUBRIFICANTE				6184-16
SEPARAZIONE DI OLIO DA GRASSI DURANTE LO STOCCAGGIO				1742-06 (2013)
SFORZO DI SOGLIA E VISCOSITÀ APPARENTE (A BASSA TEMPERATURA)				4684-14
SOLFONATI NATURALI E SINTETICI (HPLC)				3712-05 (2011)
SPECIFICA DI LIQUIDI SILICONICI PER USI ELETTRICI	60836:2015 60944:1988	CEI EN 60836:2016		
SPECIFICA DI OLI MINERALI ISOLANTI	60296:2012	CEI EN 60296:2013		
SPECIFICA PER CAPILLARI VISCOSIMETRICI	**3105:1994**	UNI ISO 3105:2001	71S2/95(04)	446-12
STABILITÀ AL ROTOLAMENTO DI GRASSI		UNI 20018:1989		1831-11
STABILITÀ ALL'OSSIDAZIONE DI OLI MINERALI INIBITI PER TURBINE		UNI 20019:1989	280/99(11)	
STABILITÀ ALL'OSSIDAZIONE DI LIQUIDI ISOLANTI NUOVI A BASE IDROCARBURI	61125:1992 am1:2004	CEI EN 61125/97+ A1:2005		
STABILITÀ ALL'OSSIDAZIONE DI OLI PER TURBINE A VAPORE (BOMBA)				2272-14a
STABILITÀ IDROLITICA DI OLI IDRAULICI				2619-09 (2014)
STABILITÀ TERMICA (in ASKAREL)	60588:1979	CEI 10-6:1997		
TEMPERATURA DI POMPABILITÀ OLIO MOTORE				3829-14
TENDENZA A FORMARE DEPOSITI E CORROSIONE				4310-10(2015)
TENSIONE DI SCARICA LIQUIDI ISOLANTI	60156:1995	CEI EN 60156:1998		
TENSIONE INTERFACCIALE DI OLI (METODO RING)	~~**6295:1983**~~			971-12
TRAFILAMENTO DI GRASSI NEI CUSCINETTI		UNI 20054:1993		~~1263-94 (2005)e1~~
CARATTERISTICHE ANTIUSURA DI GRASSI LUBRIFICANTI (MACCHINA TIMKEN)				2509-14
CARATTERISTICHE ANTIUSURA DI GRASSI LUBRIFICANTI (MACCHINA 4 SFERE)				2266-01(2015)
CARATTERISTICHE ANTIUSURA DI OLI LUBRIFICANTI (MACCHINA 4 SFERE)				4172-94 (2016)
USURA DI OLI IDRAULICI				4998-13

PARAMETRO ANALITICO	*EN*-ISO-IEC	Metodi Italiani	IP	ASTM D
USURA DI PELLICOLE SOLIDE DI LUBRIFICANTE				2981-94 (2014)
USURA E ATTRITO (MACCHINA FALEX)				2714-94 (2014)
PROPRIETÀ EP DI GRASSI (MACCHINA SRV)				5706-11
PROPRIETÀ EP DI OLI LUBRIFICANTI (MACCHINA TIMKEN)				2782-02 (2014)e1
VISCOSITÀ CINEMATICA	**3104:1994/ Cor 1:1997**	UNI EN ISO 3104 :2000	71S1/97	445-15a
VISCOSITÀ /TEMPERATURA, DIAGRAMMA				341-09(2015)
VISCOSITÀ AD ALTI GRADIENTI				4683-13
VISCOSITÀ APPARENTE DI GRASSI				1092-12
VISCOSITÀ APPARENTE DI OLI MOTORE (CCS)				5293-15
VISCOSITÀ DI LUBRIFICANTI TRAZIONE (BROOKFIELD)		UNI 20028:1992		2983-15
VISCOSITÀ DI OLI TURBINA DOPO PERMANENZA A BASSA TEMPERATURA				2532-14
VISCOSITÀ/TEMPERATURA DI OLI A BASSA TEMPERATURA, RELAZIONE				5133-15
ZOLFO (BOMBA)				129-13
ZOLFO (FLUORESCENZA RAGGI X)	**8754:2003**			4294-16e1
ZOLFO (METODO AD ALTA TEMPERATURA CON RIVELAZIONE IR)				1552-16
ZOLFO (METODO WICKBOLD)	~~**4260:1987**~~ **6326-1:2007**			
ZOLFO (FLUORESCENZA UV)				5453-16e1
ZOLFO ATTIVO DI OLI DA TAGLIO				1662-08 (2014)
ZOLFO CORROSIVO DI OLI ISOLANTI	62535:2008	UNI 20052:1992	315/98(04)	1275-15

TABELLA LUBRIFICANTI - COMMENTO ALLE NUOVE REVISIONI DEI METODI ASTM (Dicembre 2016)

PARAMETRO ANALITICO	ASTM D	COMMENTO
ACQUA IN PRODOTTI PETROLIFERI (KF)	6304-16e1	**6304-16**: modifiche alla sezione 16 Report per come esprimere l'unità di misura. **6304-16e1**: introdotta Nota editoriale per la rimozione della sezione Sommario dei cambiamenti Equivalente all'edizione 2007.
CLORO NEGLI OLI GREZZI	4929-16	Nella sezione 1 Scopo cambiato il termine "method" in "procedure". Rivista per la stessa ragione la sottosezione 25.1. Aggiunta la sottosezione 23.3 e l'Appendice X2 riguardante le norme di cautela nella preparazione di campioni da greggio tramite lavaggio con acqua. Equivalente all'edizione 2015a.
CLORO (METODO DI DECOMPOSIZIONE AD ALTA PRESSIONE)	808-16	Nella sezione 2 inserito il riferimento all'ASTM D4177 per il campionamento automatico. In 7.1 viene indicato il riferimento a questo metodo. Introdotto in 6.9 un nuovo reagente (indicatore: soluzione rosso metile). Equivalente all'edizione 2011.
DENSITÀ O DENSITÀ RELATIVA DI LIQUIDI REFRIGERANTI	1122-16	In 5.3 cancellata la frase che precisava che i dati erano ottenuti solo con l'uso di termometri a mercurio. Aggiunta Nota 2 per esplicitare che i dati di precisione e bias sono generati con la versione precedente del metodo, che prevedeva l'uso di termometri a mercurio. Equivalente all'edizione 2013.
DILAVAMENTO CON ACQUA DI GRASSI	1264-16	Aggiunta Nota 2 per i dettagli sulla pulizia dell'apparecchiatura, soprattutto per i grassi con water washout >15%. Equivalente all'edizione 2012
DILUIZIONE BENZINA DI OLIO USATO (DISTILLAZIONE)	322-97(2016)	**322-97(2012)e1**: modifiche editoriali alla sottosezione 9.4 per i tempi di lettura espressi in minuti. Cancellato il riferimento IP dal titolo e nella sezione 11 Report. Riapprovato nel 2016. Equivalente all'edizione 97(2012).
DISTILLAZIONE A PRESSIONE ATMOSFERICA DI PRODOTTI PETROLIFERI E LIQUIDI COMBUSTIBILI	86-16a	Aggiunta sottosezione 10.11.1 per spiegare il gradiente medio uniforme di condensazione. Equivalente all'edizione 2016.
ELEMENTI DI USURA E CONTAMINANTI IN OLI LUBRIFICANTI USATI O FLUIDI IDRAULICI USATI	6595-16	Nella sezione 2 inserito il riferimento all'ASTM D4177 per il campionamento automatico. In 9.1 viene indicato il riferimento a questo metodo. Introdotto in Materiali e reagenti la sottosezione 8.9 per i campioni da utilizzare per il controllo di Qualità. Equivalente all'edizione 00(2011).
MASSA VOLUMICA (DENSIMETRO DIGITALE)	4052-15	Rivista la Tabella 1 i valori di densità dell'acqua alle diverse temperature. Aggiornata in tutto il metodo l'unità di misura della pressione barometrica da torr a kPa. Sostituita alla sezione 7 "redistilled water" con " reagent water". Equivalente all'edizione 2011.
NUMERO ACIDITÀ E BASICITÀ (TITOLAZIONE CON INDICATORE)	974-14e2	Introdotte correzioni editoriali alle sottosezioni 15.1.1 e 15.1.2 (corretto acid base number con acid or base number). Equivalente all'edizione 2014e1.

PARAMETRO ANALITICO	ASTM D	COMMENTO
NUMERO DI ACIDITÀ (TITOLAZIONE POTENZIOMETRICA)	664-11ae1	Introdotta correzione editoriale alla sottosezione 14.5 (corretto base number con acid number). Equivalente all'edizione 2011a.
NUMERO DI BASICITÀ (TITOLAZIONE POTENZIOMETRICA CON ACIDO PERCLORICO)	2896-15	Aggiornata la sottosezione 10.3 per la procedura di lavaggio dell'elettrodo. Rivista a tal proposito anche la sottosezione 12.7. Equivalente all'edizione 2011.
NUMERO DI PRECIPITAZIONE PER LUBRIFICANTI	91-02(2012)e1	Introdotta correzione editoriale alla sottosezione 5.2 e alla Tabella 2 (sostituito il termine rpm con r/min). Equivalente all'edizione 02(2012).
PENETRAZIONE DI GRASSI CON CONO	217-16	Revisione fatta per aggiornare le unità di misura al Sistema Internazionale in tutto il testo e nelle figure. Equivalente all'edizione 2010.
PENETRAZIONE DI PARAFFINE CON AGO	1321-16a	**1321-16**: aggiunta sottosezione 6.7.1 per elencare diversi strumenti per la misura della temperatura. Rivista la sezione 10: aggiunte le equazioni per calcolare ripetibilità e riproducibilità di analisi condotte a 40°C e pertanto rivista la Tabella 1. **1321-16a**: rivista la sottosezione 7.1 per la preparazione del campione. Aggiunta Nota 4 e rinumerate le note. Equivalente all'edizione 10(2015).
PERDITA PER EVAPORAZIONE DI OLI E GRASSI	972-16	Rivista la sezione 2 Documenti di Riferimento per la norma E2251(caratteristiche termometri) e di conseguenza la sottosezione 6.4. Equivalente all'edizione 02(2008).
PUNTO DI CONGELAMENTO DI FLUIDI REFRIGERANTI PER MOTORI	1177-16	Nella sottosezione 6.1.2 aggiunta la parola "resistance" davanti a "thermometer". Cancellata la Nota 7 e la frase è stata inserita nella sottosezione 8.3. Rivista la prima frase della sottosezione 9.1.1. Equivalente all'edizione 2012.
PUNTO DI EBOLLIZIONE DI FLUIDI REFRIGERANTI PER MOTORI	1120-16	Rimossa la frase in 4.4 che informava che i dati di precisione sono ottenuti con l'uso di termometri a mercurio. Questa dichiarazione è stata inserita nella Nota 3. Equivalente all'edizione 2011e1.
PUNTO DI GOCCIOLAMENTO DI GRASSI	566-16	Aggiunta sottosezione 1.4 Warning con riferimento alle norme di sicurezza per l'uso di termometri a mercurio. Equivalente all'edizione 02(2009).
PUNTO DI INFIAMMABILITÀ IN VASO APERTO CLEVELAND	92-16b	**92-16**: Sezione 3 Terminologia: tolto il riferimento a prodotti petroliferi. **92-16a**: aggiornate sottosezioni A2.1.1 e Tabella A2.1 modificando l'accettabilità dei valori di CRM che per effetto delle prove interlaboratorio viene calcolata come: 0.7 × Riproducibilità del metodo. **92-16b**: inserite Nota 11 e 19 per indicazioni sul riempimento della coppa di prova. Equivalente all'edizione 2012b.
PUNTO DI INFIAMMABILITÀ IN VASO CHIUSO (PENSKY MARTENS)	93-16a	**93-16**: Sezione 3 Terminologia: tolto il riferimento a prodotti petroliferi. **93-16a**: aggiornate sottosezioni A4.1.1 e Tabella A4.1 modificando l'accettabilità dei valori di CRM che viene calcolata come 0.7 × Riproducibilità del metodo. Aggiornata sottosezione 8.2 per quanto riguarda il campionamento. Equivalente all'edizione 2015a.

PARAMETRO ANALITICO	ASTM D	COMMENTO
PUNTO DI INFIAMMABILITÀ TAG (chiuso)	56-16a	**56-16**: Sezione 3 Terminologia: aggiornata la definizione di flash point. **56-16a**: aggiornate sottosezioni A2.1.1 e Tabella A2.1 modificando l'accettabilità dei valori di CRM che per effetto delle prove interlaboratorio viene calcolata come 0.7 × Riproducibilità del metodo. Equivalente all'edizione 05(2010).
PUNTO DI INTORBIDAMENTO (RAFFREDDAMENTO LINEARE)	2500-16a	**2500-16**: Rivista la sezione 2 Documenti di riferimento. Aggiunta la sottosezione 3.1.4 per la definizione del DTC: termometro digitale di contatto. Riviste le sottosezioni 6.2, 6.4, 8.3. Rivisti i dati di precisione per la matrice biodiesel e la Nota 8. **2500-16a**: Rivisto il titolo. Rivista la sottosezione 3.1.3 sostituendo "hydrocarbon crystals" con "wax crystals". Non equivalente all'edizione 2011 per i prodotti carburanti. Equivalente all'edizione 2011 per i prodotti petroliferi.
PUNTO DI SCORRIMENTO	97-16	Alla sezione 6.2 aggiunta Nota 5 a piè di pagina per reperire i dati di supporto ai Requisiti del Termometro Digitale di Contatto (Research Report: RR:D02-1826). Equivalente all'edizione 2015.
SCHIUMEGGIAMENTO DI OLI	892-13e1	Introdotta correzione editoriali nell'Annesso 12.1. Equivalente all'edizione 2013.
SEPARAZIONE DI OLIO DA GRASSO LUBRIFICANTE	6184-16	Rivista l'introduzione. Aggiornata la sottosezione 2.2. Cancellata la Nota 1: disponibilità di dati per la comparazione con l'ASTM D1742. Equivalente all'edizione 2014.
PROPRIETÀ EP DI OLI LUBRIFICANTI (MACCHINA TIMKEN)	2782-02(2014)e1	Nota editoriale: cancellazione del logo IP e correzione della Nota 1 di fondo pagina. Equivalente all'edizione 02(2014).
VISCOSITÀ CINEMATICA	445-15a	Rivista la sezione 17 Precision and Bias ai punti riguardanti le apparecchiature automatiche-automatizzate. Cancellata la Tabella 2, riguardante i dati di precisione per strumenti automatizzati/automatici, che sono disponibili nel Research Report RR: D02-1820. Equivalente all'edizione 2015.
VISCOSITÀ DI LUBRIFICANTI TRAZIONE (BROOKFIELD)	2983-15	Rivisto completamente tutto il metodo. Non equivalente all'edizione 2009.
ZOLFO (FLUORESCENZA RAGGI X)	4294-16e1	**4294-16**: aggiornate le sottosezioni 16.1, 16.1.1, 16.1.2,16.1.3 e le appendici X1 e X2 (dati di precisione addizionali per gasolio e benzina). **4294-16e1**: correzione editoriale a tutto il layout delle sezioni dell'Appendice. Equivalente all'edizione 2010.
ZOLFO (METODO AD ALTA TEMPERATURA CON RIVELAZIONE IR)	1552-16	Rivisto completamente tutto il metodo per includere le procedure A e B. Non equivalente all'edizione 2015.
ZOLFO (FLUORESCENZA UV)	5453-16e1	**5453-16**: riviste le sottosezioni 5.1, 5.2, 5.3, 5.6, 5.7.1, 6.2 e 6.3 (modificate: l'apparecchiatura, le temperature della fornace, numero dei bracci laterali di combustione, numero di flussometri e siringa / guida dell'iniezione). Aggiunte nuove sottosezioni 6.2.1, 6.2.2 e 6.11 (aggiunti i reagenti: aria e triossido di tungsteno). **5453-16e1**: aggiunta nota editoriale per correzioni alla sottosezione 1.1. Non equivalente all'edizione 2012.

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Short note

Chemical composition of *Nabali* Jordanian olive oil

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This study covers the chemical and physical properties of the Jordanian *Nabali* virgin olive oil either commercially pressed (n = 6) or experimentally pressed (n = 6) of the harvesting season 2014 from three Jordanian olive production areas (Middle, North and South). *Nabali* olive fruit, the most popular olive variety in Jordan, could be considered of medium fruit size. No clear effect for the production area on the fruit diameter was observed, whereas, the fruit length was affected and olive fruits harvested from the North area were characterized with a significant ($p \leq 0.05$) larger length. The obtained results showed that only 3 virgin olive oil samples (25%) have free acidity that does not differ significantly ($p \leq 0.05$) from IOC standard, whereas 68% of the samples had acidity values of ≤ 2%. Such increase in free acidity results is an indication for the poor handling during processing. Peroxide values of the studied virgin olive oil samples, presented a mean value of 12.10 meq O_2 kg^{-1} and a range between 5.37 - 20.48 meq O_2 kg^{-1}. According to the obtained total phenols (TP) results, it can be concluded that the Jordanian *Nabali* virgin olive oil might be characterized with a low to moderate TP content. The olive planting area had no clear effect on the TP content of the *Nabili* virgin olive oil whereas the commercial olive mills produced oils with higher TP compared to those produced using experimental mills. The studied Jordanian *Nabali* virgin olive oil samples could be considered as having between a moderate to high level of oleic acid (65.35 - 73.05%).

Keywords: virgin Olive oil, chemical properties, fatty acid profile, total phenol content.

INTRODUCTION

Olive is considered one of the most important crops in Jordan with an annual production of about 250,000 tons of olive fruits and 35 000 tons of olive oil [1]. Jordan ranks the tenth on the list of the world's nations for olive oil production [2].

Free fatty acids are considered a vital quality parameter of olive oil. IOC standard [3] dictates that extra virgin olive oil should have free acidity of less than 0.8%, while for virgin olive oil ≤ 2% and for ordinary olive oil ≤ 3%.

Free fatty acids test is simple and the obtained figures are affected by many factors such as harvesting and handling, processing conditions, infestation by olive fly as well as others [4].

Peroxide value is a test that measures the level of primary oxidation that has occurred in the oil and it is related inversely to its stability [4]. Many factors affect peroxide value such as exposure to O_2, heat, light, metals as well as harvesting and handling processes. IOC standard for peroxide value of extra virgin olive oil is set at ≤ 20 meq. O_2 kg^{-1} oil [3].

The fatty acid profile is an important indicator of the olive oil quality and it af-

fects both nutritional value and stability; it is considered as a measure of the proportions of individual fatty acids in the oil. The IOC standards for fatty acid profile [5] dictates that myristic acid should be ≤ 0.5%, palmitic acid in the range of 7.5 - 20%, stearic acid 0.5 - 5%, oleic 55 - 83%, linoleic acid 3.5 - 21%, linolenic acid ≤ 1% and arachidic acid ≤ 0.6%. It was found [4] that an inverse proportional relationship may exist between oleic and linoleic acids. Olive oil with high oleic acid is nutritionally preferable and is more stable. However, climate, season and variety influence the fatty acid profile. It was observed that low temperature and high altitude environments produce higher oleic acid levels than high temperature and low altitude environments [4].

Among olive oil minor fraction, the phenolic compounds were reported to play an important role in olive oil oxidative stability and its organoleptic properties. It has been noticed that their content in olive oil depends on the olive variety, the harvesting time, and the climate. Besides, the optimization of olive oil storage conditions such as packaging materials, temperature, and light is of great importance to preserve phenolic compounds and their antioxidant activity. Naz [6] reported that the exposure of olive oil to heat, oxygen and light caused an alteration to its composition of especially fatty acid and antioxidant contents, resulting in a lowering of the health benefits. Large variations were reported in literature for olive oil phenolic content; values ranging between 60 and 400 ppm were given by the researchers [4, 7, 8]. Due to the little information available in literature regarding Jordanian olive oil composition, the scope of this work is to establish the chemical characterization of Jordanian olive oils. In 2014, a multi-country project titled “Capacity building of personnel in Jordanian olive industry” was initiated. The project is funded by the Executive Agency Education Audiovisual and Culture [EACEA] of the European Union; it is coordinated by the University of Jordan and has other 9 institutions as partners, of which 5 are national and four from the EU. The main objective of this project is to improve the quality of Jordanian olive oil.

2. MATERIALS AND METHODS

2.1 OLIVE AND OLIVE OIL SAMPLING AND PRESSING

The experiment was designed to include 6 olive samples from the *Nabali* variety, representing the three major olive producing areas in Jordan. Olives of the *Nabali* cultivar were harvested in November 2014 (15 kg for each batch) in two orchards located in the north (Irbid and Ajloan), two in the south (Karak and Alhusainyah) and two in the center (Salt) Jordan. Olives were stored at room temperature (15 - 20°C) for a maximum duration of 48 hrs. The olive fruits were milled at the set temperature (28 - 32°C) by centrifuge with an experimental Olimio Mini 50 type press with a capacity of 50 kg/hr. Another 6 olive oil samples representing the above 3 Jordanian olive producing areas were taken from commercial olive mills. The samples were filled into brown glass bottles and stored in darkness until the analysis took place.

2.2 ANALYTICAL DETERMINATION OF THE QUALITY INDICES

The mean average of the length and diameter of 30 fresh olive fruits for each olive sample was measured for the nearest mm using the venires caliber. Free acidity and peroxide value were determined according to the EC regulation n° 2568/91 and the following amendments [9].

2.3 FATTY ACID COMPOSITION

Fatty acid composition was determined following the EC Regulation n° 2568/91 [9]. Fifty mg of lipid extract was weighed, dissolved in 1 ml hexane (GC grade) and mixed by vortex for 1 min. A 200 µl of 2 M-potassium hydroxide prepared in anhydrous methanol was added and mixed for 30 sec until the solution became clear. The prepared methyl esters (FAMEs) were analyzed using capillary GLC column (Restek, Rtx-225, USA, cross-bond 50%-cyanopropylmethyl 50%-phenylmethylpolysiloxane, 60 m, 0.25 mm/D, 0.25 µmdf) immediately after esterification by injecting 1 µl of the hexane layer through the injection port of the GLC (model GC-2010, Shimadzu. Inc., Koyoto, Japan). The FAMEs were injected after adjusting the GLC conditions; column oven temperature was 180°C for 10 min, increased to 200°C 5°C/min and kept at 200°C for 5 min, then increased to 210°C 3°C/min and kept at 210°C for 20 min. Injector temperature was 250°C, the flame ionization detector temperature was 260°C, flow rate 1.2 ml/min N2, and split ratio used was 70. The fatty acids methyl esters (FAMEs) were identified using the chromatogram of fatty acids standard.

2.4 PHENOLIC CONTENT

Total phenols content were measured in the polar fraction extracted twice from 15 g olive oil using methanol-water mixture (60:40 v/v); the combined extracts were layout to dryness using rotary evaporator at 50°C. The residue was dissolved in 100 ml methanol.

Samples of 1ml of the methanol solution were transferred into a 10 ml test tube and the 3 ml volume was reached with distilled water. An amount of 0.5 ml of FCR was added and mixed well. After 3 min, 2 ml of 20% (w/v) sodium carbonate solution (20% w/v) were added. The solution was left to stand for 60 min and the absorbance of the sample was then measured at 720 nm using a spectrophotometer (Biotech Engineering Management Co., UK). The concentration of the total phenolic compounds was calculated by comparison with the absorbance of different concentrations of gallic acid and the total phenolic compounds content of the plant extracts was expressed as gallic acid equivalent [10].

Table I - Statistical parameters of chemical and physical properties of *Nabali* Jordanian olives and olive oils

Statistical parameters	Free Acidity % as oleic acid	Peroxide Values meq.O2/kg	Total Polyphenols mg/kg	Fruit Diameter (cm)	Fruit Length (cm)
Mean	1.49	12.10	158.19	1.56	2.17
LSD	0.11	1.11	5.41	0.21	0.37
SD	0.07	0.66	3.21	9.122	0.22
CV	4.48	5.47	2.03	7.84	10.10
Range	0.88 - 2.58	5.37 - 20.48	101.15 - 281.50	1.25 - 1.77	1.97 - 2.36
IOC*	0.80	20	-	-	-

(*) IOC: International olive oil standards

2.5 STATISTICAL ANALYSIS

Data were analyzed using one-way analysis of variance (SAS, 2004) [11]. Means were compared using LSD statistical tool. Significance was set at ($p \leq 0.05$). The means for each sensory olive oil attribute was also calculated using the IOC statistical computer program (IOC, 2007) and the classification of the oil was determined accordingly.

3. RESULTS AND DISCUSSION

Results of quality indices for commercially pressed (n = 6) and experimentally pressed (n = 6) *Nabali* olive oil samples of the harvesting season 2014 from three Jordanian olive production areas (Middle, North and South) are shown in Tables I and II and Figures 1-5. Table I reports mean values, standard deviations range, least significant differences, coefficient of variation

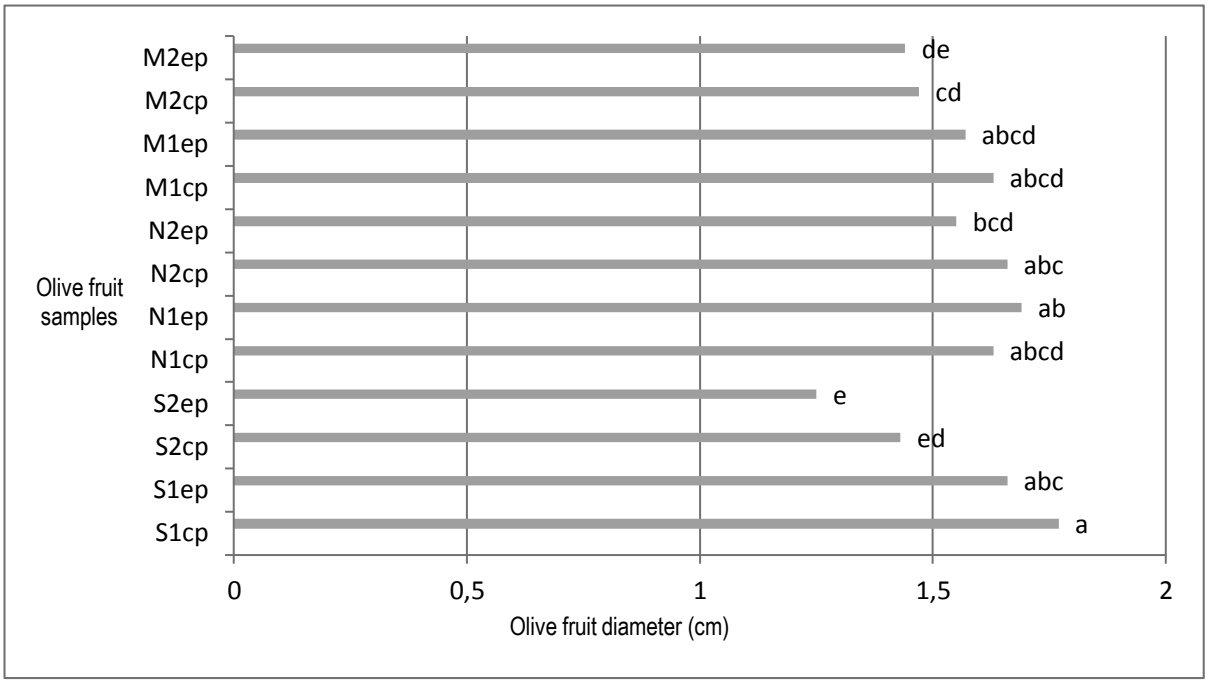

Figure 1 - Fruit diameter of *Nabali* Jordanian olives from 3 production areas
S1cp: The 1st olive oil sample taken from the south of Jordan and extracted by commercial press
S1ep: The 1st olive oil sample taken from the south of Jordan and extracted by experimental press
S2cp: The 2nd olive oil sample taken from the south of Jordan and extracted by commercial press
S2ep: The 2nd olive oil sample taken from the south of Jordan and extracted by experimental press
N1cp: The 1st olive oil sample taken from the north of Jordan and extracted by commercial press
N1ep: The 1st olive oil sample taken from the north of Jordan and extracted by experimental press
N2cp: The 2nd olive oil sample taken from the north of Jordan and extracted by commercial press
N2ep: The 2nd olive oil sample taken from the north of Jordan and extracted by experimental press
M1cp: The 1st olive oil sample taken from the middle of Jordan and extracted by commercial press
M1ep: The 1st olive oil sample taken from the middle of Jordan and extracted by experimental press
M2cp: The 2nd olive oil sample taken from the middle of Jordan and extracted by commercial press
M2ep: The 2nd olive oil sample taken from the middle of Jordan and extracted by experimental press

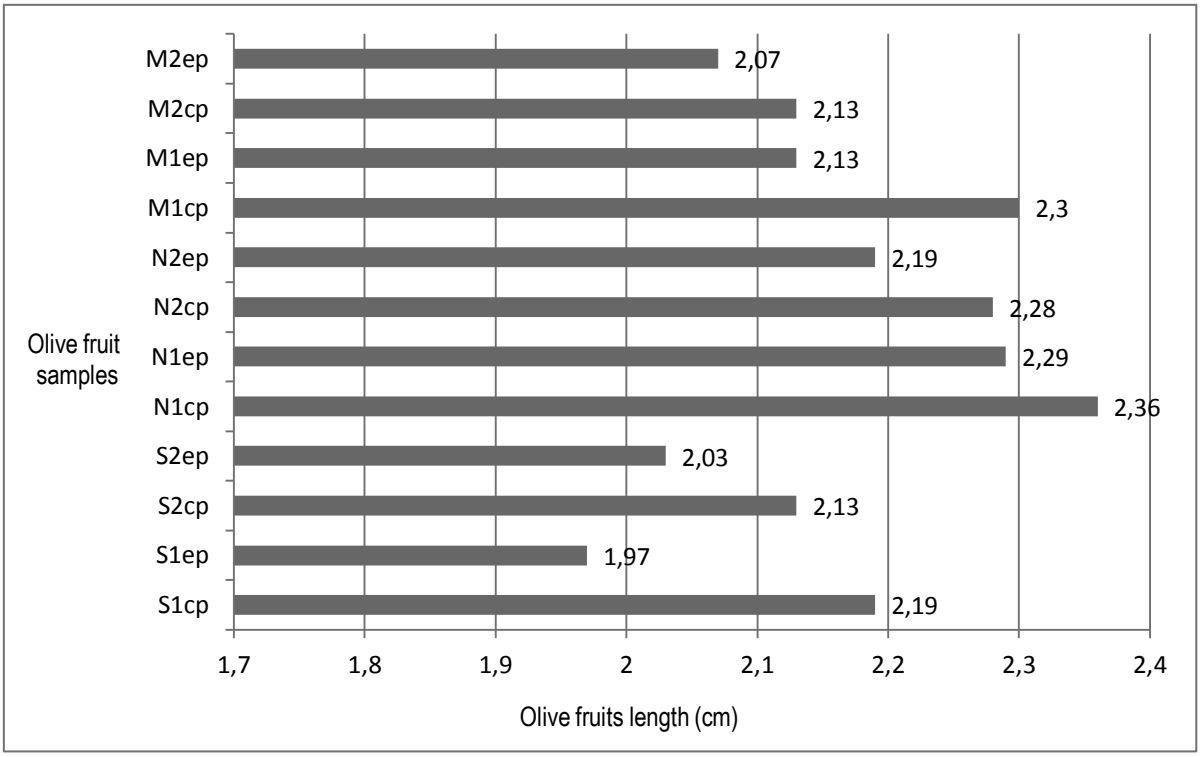

Figure 2 - Fruit length of *Nabali* Jordanian olives* from 3 production areas
Names of olive fruit samples as in Figure 1.

as well as IOC standards for olive oil free acidity, peroxide values and total phenolic compounds contents.

3.1 OLIVE FRUIT DIAMETER AND LENGTH

It is clear from the results in Table I and Figure 1 that *Nabali* olive fruit diameter and length ranged from 1.25 up to 1.77 and from 1.97 up to 2.36 cm respectively. These results indicated that *Nabali* olive fruit, the most popular olive variety in Jordan, could be considered of medium fruit size. No clear effect for the production area on the fruit diameter were observed (Fig. 1). However, the fruit length was affected by the production area (Fig. 2) and olive fruits harvested from the North area were characterized with a significant ($p \leq 0.05$) larger length. These results might be ascribed to several factors of which the annual precipitation (for the 2014 season, 200, 400 and 450 ml for south, north and middle areas were estimated respectively) and pre-harvest operations are the most important.

3.2. FREE ACIDITY

Increased free acidity is generated by the degradation that takes place in the oil through poor handling during processing [4]. It is clear from results in Table I, that the acidity mean for the studied samples reached 1.49% as oleic acid. The free acidity values ranged between 0.88 up to 2.58% with a standard deviation of 0.11 and a coefficient of variation of 4.48. Such statistics indicate an acceptable accuracy of the obtained results.

Results of Fig. 3 show that only 3 olive oil samples (25%) have free acidity that does not differ significantly ($p \leq 0.05$) from IOC standard, whereas 68% of the samples had acidity values minor than 2%. Furthermore, one sample (8.5%) showed a free acidity value higher than 2% but less than 3% (2.58%). However, no consistent effect for the production area or the type of press (commercial or experimental) on the free acidity results was established. The achieved free acidity values for the studied olive oil samples were higher than those reported by Al-Ismail [12] who studied the effect of reclaimed water on the chemical properties of Jordanian olive oils where lower figures were reported (0.11 - 0.29%). Additionally, lower free acidity values (0.23 - 0.44%) were reported by Alsaed who studied the effect of packaging material on the quality of Jordanian olive oil [13]. However, the obtained free acidity values are relatively close to those (0.15 - 1.83%) reported for Spanish *Cornicabra* olive oil [14]. Finally, such relatively increased free acidity results obtained in this study is an indication for the poor handling during processing. As a result, good handling of olive fruits during harvesting, transportation, storage and pressing as well as storage of olive oil is considered of vital importance to produce Jordanian olive oil that conform to the IOC standards and can compete in the global market.

3.3 PEROXIDE VALUE

Peroxide value, expressed as meq O_2 per kg, presented a mean value of 12.10 and a range between 5.37 - 20.48. Only 1 sample (8.5%) had a peroxide

value higher than the upper limit of 20 established for extra virgin olive oil by IOC. As in FA, the production area did not show any consistent effect on the peroxide values of the studied olive oil samples (Tab. I and Fig. 4). Nevertheless, the press type had a significant effect on the peroxide value; experimental pressing (S1ep, S2ep, N1ep, N2ep and M1ep) resulted in olive oils with significant ($p \leq 0.05$) lower peroxide values.

It is evident from Table I that the obtained peroxide values had a standard deviation of 0.66 and coefficient of variation of 5.47 that indicates an acceptable accuracy for the obtained results.

The achieved peroxide values for the studied olive oil samples were close to those reported for Spanish *Cornicabra* olive oil (3.60 - 29.60) [14] and for Turkish olive oils (7.37 - 22.30) [15] as well as for Jordanian

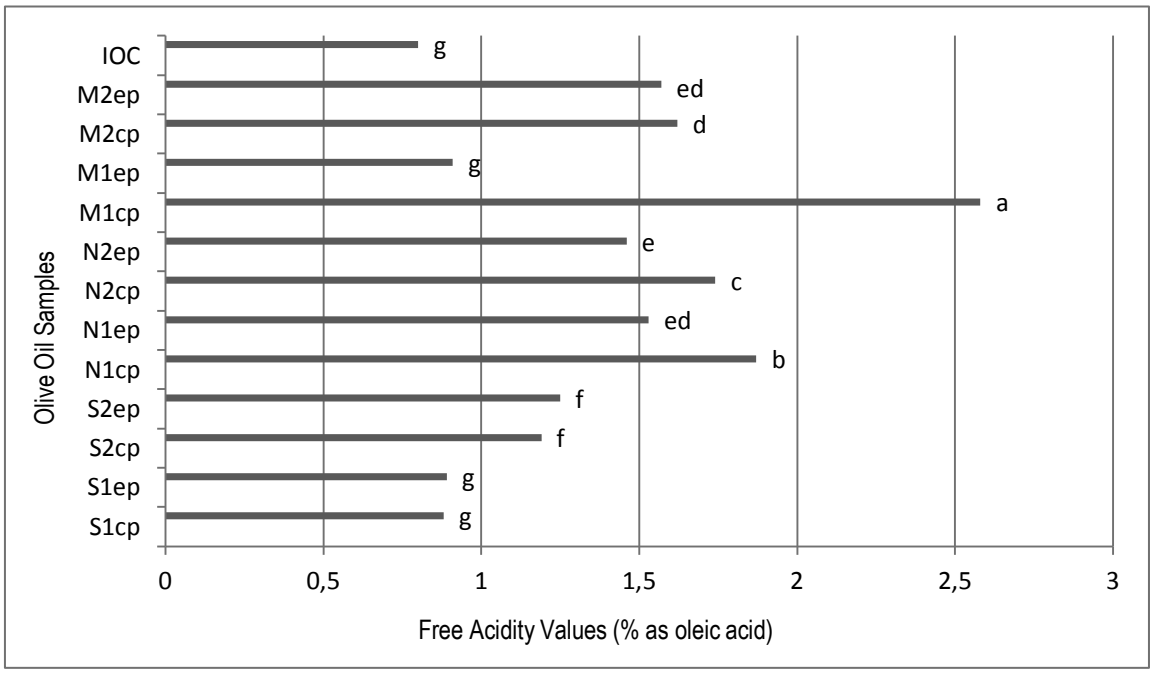

Figure 3 - Free Acidity values of Jordanian olive oil samples* taken from 3 olive producing areas and pressed by 2 methods
Names of olive oil samples as in Figure 1.

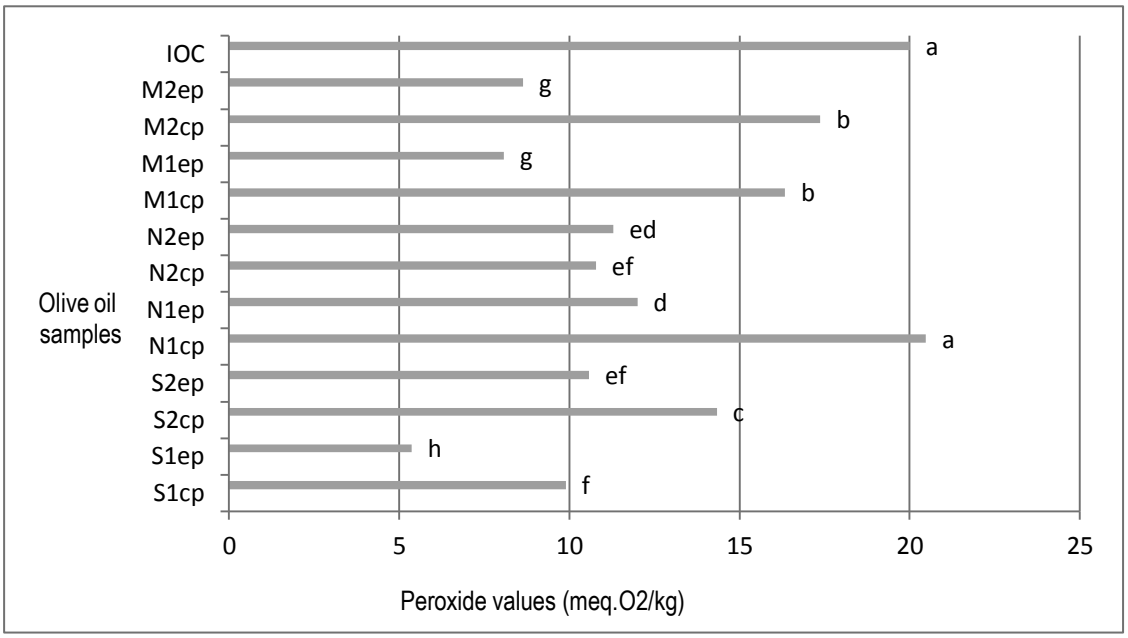

Figure 4 - Peroxide values of Jordanian olive oil samples* taken from 3 olive producing areas and pressed by 2 methods
Names of olive oil samples as in Figure 1.

olive oil samples (12 - 21.30) that were reported by Alsaed who studied the effect of packaging material on the quality of Jordanian olive oil [13]. However, the obtained peroxide values were higher than those reported by Al-Ismail [12] who studied the effect of reclaimed water on the chemical properties of Jordanian olive oils for which lower data were reported (0.90 - 2.5). Lastly, such relatively increased peroxide results obtained in this study is an indication for the poor handling during processing, especially when using commercial mills. As a result, good handling during commercial pressing is considered important to produce Jordanian olive oil that conforms to the IOC standards and can compete in the global market.

3.4. TOTAL POLYPHENOLS

The mean of the total content (TP) in the Jordanian *Nabali* olive oil samples analyzed was 158.19 mg/kg with a standard deviation of 3.21, coefficient of variation of 2.03% (Tab. I and Fig. 5). However, a relatively broad and significant ($p \leq 0.05$) variation could be observed in the obtained TP results (101.15 - 281.50 mg/kg). Only 1 olive oil sample (8.5%) obtained from a commercial mill from the North of Jordan had a TP concentration higher than 200 mg/kg. Six samples (50%) had a TP concentration of higher than 150 mg/kg; 3 of those samples were from the south, 2 from the north and 1 from the middle of Jordan, 4 of those 6 samples were obtained from a commercial mill. Such results might refer to the absence of a strong effect for the olive planting area on the TP content of olive oil while such effect exists for the type of mill.
It was reported [4] that TP are not essential for high quality olive oil. Delicate fruity oils with very low TP levels can be reasonably stable and highly marketable. Olive oils with moderate levels of TPP (200 - 400 mg/kg) can be easier to manage than very strong flavored oils. Alsaed [13] reported higher values (235 mg/kg) for TP in Jordanian *Nabali* olive oils, whereas Al-Ismail [12] reported almost similar TP values (106.5 - 193.2 and 125.7 - 247.7 mg/kg) for Jordanian *Nabali* olive oils of 2006 and 2007 harvesting years respectively. TP ranging from 19 to 380 mg/kg for Spanish Cornicabra virgin olive oil and from 18.7 - 242.5 mg/kg for Greek virgin olive oil was reported by Salvador [14] and Tsimidou [7], respectively.
According to the obtained TP results, it can be concluded that the Jordanian *Nabali* olive oil might be characterized with a low or moderate TP content and the olive planting area had no clear effect on the TP content of the *Nabali* olive oil whereas the commercial olive mills produced oils with higher TP compared with those produced using experimental mills.

3.5. FATTY ACID COMPOSITION

The fatty acid composition of the Jordanian *Nabali* olive oil samples taken from 3 olive planting areas and produced by two types of mills i.e commercial and experimental for the 2014 harvesting season is shown in Table II. The distribution of the fatty acid composition of the studied *Nabali* olive oil samples cover the normal range expected for olive oil and are within the permitted level as mentioned by the International Olive Council [5]. Higher values for palmatic, linoleic, and linolenic fatty acid and lower one for stearic, and oleic fatty acids of the Jordanian *Nabali* olive oil were

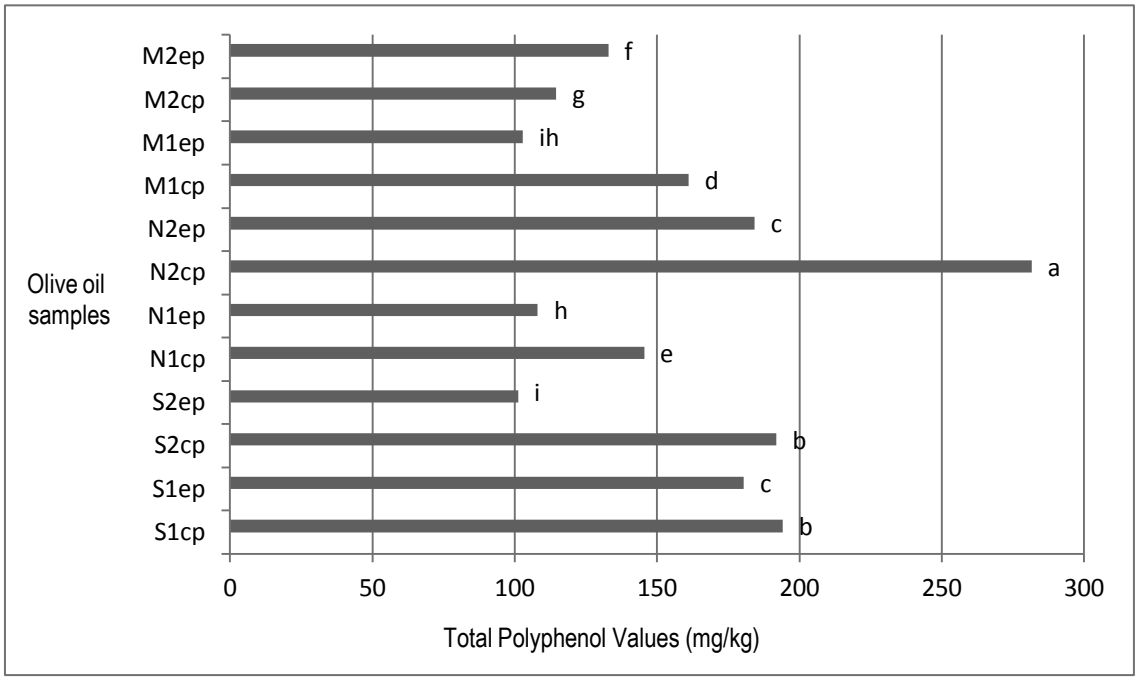

Figure 5 - Total phenol values of Jordanian olive oil samples* taken from 3 olive producing areas and pressed by 2 methods
Names of olive oil samples as in Figure 1.

Table II - Fatty acid composition (%) of *Nabali* Jordanian olive oils

				Fatty	Acid	Profile					
		C14:0	**C16:0**	**C16:1**	**C17:0**	**C17:1**	**C18:0**	**C18:1**	**C18:2**	**C18:3**	**C20:0**
Olive*	S1cp	0.012b	13.6bc	0.93b	0.045i	0.11ef	2.4g	71.07cb	9.87f	0.66b	0.40e
	S1pp	0.009d	15.25a	1.02a	0.095gh	0.085f	3.06ef	65.35h	13.85a	0.73a	0.46cd
Oil	S2cp	0.015a	13.3bc	0.82c	0.125gf	0.16d	3.29ed	70.01e	11.83c	0.61b	0.45cde
	S2pp	0.013ab	12.25dce	0.65d	0.155ef	0.165cd	3.36ecd	69.20f	13.10b	0.65b	0.48cb
Samples	N1cp	0.012b	11.4gfe	0.48fg	0.205cd	0.21b	3.78bc	70.57cd	11.32d	0.63b	0.52b
	N1pp	0.013ab	10.55gf	0.48fg	0.245b	0.275a	4.04ba	71.50b	11.20d	0.63b	0.53b
	N2cp	0.009d	14.35ba	0.81c	0.075ih	0.09f	2.64gf	69.25f	11.20d	0.63b	0.42de
	N2pp	0.008d	11.4gfe	0.54fe	0.33a	0.27a	4.35a	69.17f	11.80c	0.63b	0.59a
	M1cp	0.015a	15.68a	0.8c	0.155ef	0.195cb	3.41ecd	66.55g	11.76c	0.61b	0.52b
	M1pp	0.015a	12.9dc	0.57e	0.13gf	0.135ed	3.66bcd	70.36ed	10.59e	0.52c	0.50cb
	M2cp	0.012b	11.87dfe	0.49fg	0.225cb	0.25a	3.92ba	72.87a	10.15f	0.62b	0.53b
	M2pp	0.01dc	10.35g	0.43g	0.185ed	0.21b	3.94ba	73.05a	10.0f	0.53c	0.53b
	Mean	0.012	12.74	0.67	0.16	0.18	3.49	69.91	11.39	0.62	0.49
	LSD										
	SD	0.001	0.64	0.03	0.016	0.014	0.21	0.252	0.14	0.024	0.027
	CV	8.48	6.03	4.58	9.79	7.96	6.02	0.36	1.24	3.93	5.5
	Range	0.008 - 0.015	10.40 - 15.68	0.43 - 1.02	0.045 - 0.330	0.090 - 0.275	2.40 - 4.35	65.35 - 73.05	9.87 - 13.85	0.52 - 0.73	0.40 - 0.59
	IOC	≤ 0.05	7.5 - 20.0	0.3 - 3.5	≤ 0.3	≤ 0.3	0.5 - 5.0	55.0 - 83.0	3.5 - 21.0	≤ 1.0	≤ 0.6
	Standard										

(*) Names of olive oil samples as in Figure 1.

reported by Alsaed [13]. The obtained fatty acid composition for the studied olive oil samples agrees with those reported by Al-Ismail [12]. It is well known that olive oil with high oleic acid is nutritionally preferable and potentially more stable than low oleic olive oil. The IOC standard for oleic acid is between 55 - 83%, and the studied Jordanian *Nabali* olive oil samples could be considered as having between moderate to high level of oleic acid (65.35 - 73.05%).

4. CONCLUSION

Three *Nabali* olive oil samples (25%) have free acidity that does not differ significantly ($p \leq 0.05$) from IOC standard, whereas the other samples had acidity values of ≤ 2%. Such increased free acidity results is an indication for the poor postharvest handling. Peroxide values of the studied olive oil samples, presented a mean value of 12.10 which can be considered relatively high in spite of being less than the margin level of the IOC standard (20 mg equiv. O2/1kg) and again this is an indicator of the poor postharvest processes. The Jordanian *Nabali* olive oil might be characterized with a low to moderate TP content and the olive planting area had no clear effect on the TP content of the *Nabali* olive oil whereas the commercial olive mills produced oils with higher TP compared to those produced using experimental mills. The studied Jordanian *Nabali* olive oil samples could be considered as having between moderate to high level of oleic acid (65.35 - 73.05%).

Acknowledgment

The researchers thank the Education, Audiovisual and Culture Executive Agency (EACEA) of the European Union for their financing of this project.

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Received March 7, 2016
Accepted April 29, 2016

Evaluation of the suitability of *Tetracarpidium conophorum, Pentaclethra macrophylla* and *Citrullus vulgaris* seed oils for some fried products

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Frying performance of crude melon seed oil, conophor nut oil and African oil bean seed oil was investigated by assessing the physicochemical changes of the oils during frying of plantain, yam and potato chips. Sensory evaluation was conducted on the fried products using untrained panelists to assess acceptability of the oils for frying. The storage stability of the chips was also compared. The chips were fried in each oil for two minutes, packaged and stored at room temperature. Lipids were extracted from the stored chips at two-week intervals and analyzed for Acid Value, Peroxide Value, Iodine Value, Saponification Value and Free Fatty Acids. Conophor nut oil had Free Fatty Acid (FFA) values of 1.86 mg/g before frying and 10.66 mg/g after frying. Melon seed oil had FFA values of 1.02 and 2.26 mg/g before and after frying respectively. Melon seed oil fried products had the highest sensory ratings with general acceptability scores of 7.50, 7.57 and 6.60 for potato, plantain and yam chips respectively. Conophor nut oil fried products had the least general acceptability scores of 3.80, 4.20 and 3.42 for potato, plantain and yam chips respectively. Chips fried in conophor nut oil had a greater rate of accumulation of peroxide and free fatty acids over the 4 weeks of storage having peroxide values of 9.44, 35.73 and 11.96 mg/g for potato, plantain and yam fried chips respectively in the second week of storage. These increased to 13.93, 87.14 and 16.00 mg/g by the fourth week of storage. Chips fried in Melon seed oil had the least accumulation of peroxides with peroxide values of 0.73, 19.22 and 10.32 mg/g for potato, plantain and yam fried chips respectively in the second week and 12.61, 23.21 and 14.00 mg/g in the fourth week. Among all three frying oils, melon seed oil was the most stable while conophor nut oil generally exhibited the least chemical stability during frying. Conophor nut oil fried products were least accepted while melon seed oil fried products were the highest rated.

INTRODUCTION

Fried foods are far more popular today in many places and this is noticed observing e rapidly increasing number of fast food restaurants and vendors in the last few decades. Aside from their high caloric value, fried foods can be nutritious and favorably compared with other cooking methods such as baking or boiling. It is important to understand the factors affecting the deterioration of frying oil and monitor the quantity of products of decomposition to ensure the quality of fried foods. Improving oil quality, considerations on residence time and design are typical examples of frying technology that is still evolving [26].

Neglected and underutilized crops are domesticated plant species that have been used for their food, fiber, fodder, oil or medicinal properties but have been reduced in importance over time owing to particular supply and use

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constraints. Some plants have been so neglected that genetic erosion of their genepools has become so severe that they are often regarded as lost crops [31]. As the demand for plant and crop attributes changes, neglected crops can overcome the constraints to wider production and use.
Much work has been done on improving the shelf life of fried foods and providing alternatives to the commonly used oils [4]. The chemical composition of oil is a key factor in the risk of rancidity. Other variables involved in the process include frying conditions, replenishment of fresh oil, original oil quality, food materials and fryer type [23]. This study was to evaluate the suitability of Conophor nut, African oil bean and Egusi melon seed oils for frying.

MATERIALS AND METHODS

Conophor nut, African oil bean and Egusi melon seeds were purchased from Akure main market (Oja Oba) in Ondo state, Nigeria. Oils were extracted from the samples using soxhlet apparatus with 95% n-hexane as the extracting solvent. The hexane was evaporated to yield the crude oils. Plantain, potato and yam chips were fried in each oil at 185 ± 5°C for 2 minutes.
Proximate composition of samples was determined using AOAC [1] methods of analysis. Carbohydrate was determined by difference method.

MINERAL CONTENT DETERMINATION

Minerals of each sample (Ca, Mg, Zn, Fe) were determined by Flame Atomic Absorption Spectrophotometer as described by AOAC (2000). K and Na were determined using flame photometer. A weighed 0.5 g of each sample was digested in 6.5 ml of acid solution (HNO_3, H_2SO_4, $HClO_4$ in ratio of 5:1:0.5). The corresponding solution was heated until white fumes appeared. The clear solution was diluted up to 50 ml with distilled water and filtered with Watman filter paper no. 41. The standard working solutions of elements of interest were prepared to make the standard calibration curve. Absorption for a sample solution uses the calibration curves to determine the concentration of a particular element in that sample. Cathode lamps are used as radiation source. Air acetylene gas was used for all the experiments. This method provides both sensitivity and selectivity since other elements in the sample will not generally absorb the chosen wavelength and thus, will not interfere with the measurement.

DETERMINATION OF SPECIFIC GRAVITY

Specific gravity at 30°C = A-B/C
Where:
A = weight (g) of specific gravity bottle with oil at 30°C
B = weight (g) of specific gravity bottle at 30°C
C = weight (g) of specific gravity bottle with water at 30°C

Ester value determination

The Ester value is defined as the mg of KOH required to react with glycerine (or glycerol) after saponifying one gram of fat. It is calculated from the Saponification Value (SV) and the Acid Value (AV).
Ester Value (EV) =
= Saponification Value (SV) - Acid Value (AV)
Other physicochemical properties like refractive index, flash point, fire point, Acid value, saponification value, Iodine value, peroxide value and unsaponifiable matter were determined using the A.O.C.S. (2004) methods of analysis.
Sensory evaluation of fried products was carried out using the method described by Olopade *et al.* [20]. Means were tested for differences by analysis of variance (ANOVA) using Statistical Analysis Package for Social Sciences (SPSS) 17. Significant differences between mean values were determined by Duncan's multiple range test and accepted at $P \leq 0.05$.

Table I - Physicochemical properties of oils.

Parameter	Soybean Oil	Melon seed oil	Conophor nut oil	AOB Oil
R.index	1.45^{a}	1.48^{a}	1.47^{a}	1.47^{a}
Specific Gravity (g/cm)	0.87^{a}	0.91^{a}	0.89^{a}	0.97^{a}
Viscosity (CP)	44.13^{b}	46.63^{a}	33.13^{c}	36.23^{d}
Smoke point (°C)	178.50^{b}	184.00^{a}	122.00^{d}	163.00^{c}
Flash point (°C)	288.00^{b}	318.00^{a}	156.00^{d}	210.00^{c}
Fire point (°C)	319.00^{c}	336.00^{a}	202.00^{d}	287.00^{c}
Acid value (mg/g)	3.37^{b}	2.02^{d}	3.70^{a}	3.07^{c}
Iodine Value (mg/g)	159.90^{d}	177.70^{c}	276.64^{a}	206.00^{b}
Peroxide Value (mg/g)	21.00^{b}	9.40^{c}	42.00^{a}	7.00^{d}
Saponification Value (mg/g)	153.24^{c}	160.25^{b}	120.31^{d}	178.00^{a}
Free fatty Acids (mg/g)	1.69^{b}	1.02^{d}	1.86^{a}	1.54^{c}
Unsaponifiable Matter (%)	2.42^{a}	2.81^{a}	1.42^{b}	1.92^{ab}
Ester Value (mg/g)	149.87^{b}	158.23^{a}	83.33^{d}	147.33^{c}

*Values with the same letter superscripts on the same row are not significantly different at $p < 0.05$
Legend: A.O.B Oil - African Oil Bean seed oil

RESULTS AND DISCUSSION

Table I shows the physicochemical properties of the oils. The refractive indices of the oils are within the range of values obtained from previous research. Specific gravity signifies the extent of adulteration of the oil and is usually used in industry as a marker for oil quality [28]. The specific gravity of melon seed oil was higher than that reported by Oluba *et al.* [21] but the difference was not significant. The value obtained for African oil bean seed oil was also slightly higher than that reported by Odoemelam [17]. Acid value is a measure of hydrolytic rancidity occurring as a result of the native lipases in the oils. Acid value for melon seed oil was significantly lower to that reported by Egbebi [10] and Oluba *et al.* [21]. High acid value for conophor nut and African oil bean seed oils could be due to poor handling, improper storage and length of time in storage [28]. Iodine value gives a measure of saturation in the oil. Higher iodine values indicate higher degrees of unsaturation. Conophor nut oil has high iodine values making it good for soap making but less effective as a drying oil. Iodine value also show susceptibility to oxidative rancidity. AOB oil is least susceptible to oxidative rancidity. This is in agreement with the result of Talabi and Enujiugha [28].

Acid value increased in all the oils because of the formation of free fatty acids during frying and storage (Table II). Conophor nut oil showed its susceptibility to hydrolytic oxidation, having the highest acid value. The iodine value increased drastically showing higher degrees of unsaturation in the oils. Cooking oils with more saturated fatty acids such as lard and palm oil are usually more stable for frying. On the other hand, oils with more unsaturated fatty acids are less stable and decompose easily at high frying temperature [8]. Conophor nut oil contains over 60% linolenic oil [18], while African Oil Bean Seed Oil and Melon seed oil contain over 60% linoleic acid [11, 16]. The double bonds rearrange during frying. The peroxide value increased because of auto-oxidation helped along by the heat. Reduction in saponification value indicates destruction of the longer chain fatty acids, the results show that repeated use of the oils for frying would cause rancidification. Rancidification can reduce the value of food and some vitamins are highly sensitive to degradation [29].

Considering plantain and its fried products, the moisture content was reduced after frying as would be expected (Table III). Fat content was higher in Melon fried plantain. This is because of oil penetration into the food as melon seed oil has the highest smoke, flash and fire points of all the oils making it stable for prolonged frying times. Frying increases dietary fiber content in foods [12]. Fiber content for melon seed oil fried plantain was higher while conophor nut oil fried plantain had the least. Dietary fiber is important in the prevention of diseases such as diabetes and cardiovascular diseases. Protein content decreased in all fried products, this is probably due to brown-

Table II - Physicochemical properties of oils after frying

Parameters	Soybean oil	Melon seed Oil	Conophor nut Oil	AOB Oil
R. index	1.47^{a}	1.47^{a}	1.47^{a}	1.47^{a}
Viscosity(CP)	42.57^{d}	45.92^{c}	57.21^{b}	61.70^{a}
Smoke Point (°C)	148.00^{b}	142.00^{c}	138.00^{d}	160.00^{a}
Flash Point (°C)	282.00^{a}	220.00^{c}	182.00^{d}	250.00^{b}
Fire Point (°C)	330.00^{a}	300.00^{c}	210.00^{d}	310.00^{b}
Acid Value (mg/g)	3.44^{d}	4.49^{c}	21.21^{a}	12.34^{b}
Iodine Value	456.84^{b}	406.08^{c}	492.37^{a}	352.78^{d}
Peroxide (mg/g) Value(mg/g)	30.00^{c}	40.00^{b}	60.00^{a}	20.00^{d}
Saponification Value (mg/g)	218.79^{a}	75.74^{c}	74.33^{d}	183.73^{b}
Free Fatty Acids (mg/g)	1.72^{d}	2.26^{c}	10.66^{a}	6.20^{b}
Ester Value (mg/g)	216.55^{a}	71.25^{d}	137.73^{c}	171.39^{b}

*Values with the same letter superscripts on the same row are not significantly different at $p < 0.05$
Legend: A.O.B Oil - African Oil bean seed oil

Table III -Proximate composition of plantain and its fried product

Samples	Ash Content	Moisture Content	Fat Content	Crude Fibre Content	Crude Protein Content	Carbohydrate Content
Plantain	0.59 ± 0.02^{c}	50.10 ± 0.20^{a}	1.61 ± 0.20^{e}	14.95 ± 0.73^{d}	2.60 ± 0.14^{a}	30.15 ± 0.89^{a}
AOB Plantain	0.99 ± 0.04^{a}	28.97 ± 0.60^{d}	25.64 ± 0.60^{b}	18.45 ± 0.30^{c}	1.49 ± 0.08^{d}	24.46 ± 0.26^{c}
Soybean Plantain	0.34 ± 0.02^{d}	29.88 ± 0.20^{c}	21.80 ± 0.10^{d}	18.95 ± 0.19^{b}	1.86 ± 0.28^{c}	27.08 ± 0.07^{b}
Conophor Plantain	0.82 ± 0.01^{b}	40.98 ± 0.50^{b}	23.79 ± 0.08^{c}	13.90 ± 0.85^{e}	1.47 ± 0.18^{d}	19.04 ± 0.10^{d}
Melon Plantain	0.94 ± 0.05^{a}	28.59 ± 0.17^{e}	33.01 ± 0.93^{a}	19.95 ± 1.01^{a}	2.22 ± 0.24^{b}	15.29 ± 0.06^{e}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Plantain - Plantain fried in African Oil Bean seed oil; Soybean Plantain - Plantain fried in Soybean oil; Conophor Plantain - Plantain fried in Conophor nut oil; Melon Plantain - Plantain fried in Melon seed oil

ing reactions as it is supported by the corresponding decrease in carbohydrate content [15]. Mineral changes are shown on Table IV. Most minerals are non-volatile therefore the content of minerals on wet weight is expected to rise after frying [12]. In humans, sodium is an essential mineral that regulates blood volume, blood pressure, osmotic equilibrium and pH [27]. Sodium content increased in all fried products with melon seed oil fried plantain having the highest sodium content. Magnesium is important for enzymes catalyzing the synthesis of ATP and for those that use other nucleotides to synthesize DNA and RNA [25]. Magnesium content generally decreased for all fried products except melon oil fried plantain. Mineral losses in fried foods are because of oil uptake [12].

In the case of potato and its fried products, ash content was reduced by frying as shown in Table V. This is in agreement with the work of Oluwaniyi and Dosumu [22] where it was noticed that frying reduced the ash content of fried fish. The lower ash content is due to decreases in the mineral content of fried potatoes. Melon potato had the highest ash content while soybean potato had the lowest. This is in agreement with the results of the fried plantain analysis. Fat content of fried foods is expected to increase because of absorption of the oil by the food. This is supported by the previous research of Bognar [5] that showed the increases in fat content of fried potatoes compared to raw ones. Potassium cations are important in neuron (brain& nerve) function, and in influencing the osmotic balance between cells and the Campbell interstitial fluid [7]. Potassium content decreased in all fried products. Bognar [5] showed that potassium is easily lost during food processing. Diets low in potassium lead to hypertension [30]. Zinc is essential for many physiological roles in a number of enzyme actions in living systems. Zinc content increased in melon and AOB fried potatoes but decreased in soybean and conophor fried potatoes (Table VI). This is because of the varying oil penetration and moisture loss [12].

For yam and its fried products, there was an increase in ash content of fried products signifying increases in mineral content; this is in contrast with the results

Table IV - Mineral content of plantain and its fried products

Samples	Na	K	Ca	Mg	Zn	Fe
Plantain	57.63^{e}	48.13^{e}	25.52^{e}	73.23^{b}	0.67^{b}	2.24^{b}
AOB Plaintain	73.13^{c}	68^{c}	57.03^{b}	56.95^{c}	0.46^{c}	1.52^{c}
Soybean Plantain	83.01^{b}	72.08^{b}	38.28^{d}	32.54^{d}	0.3^{d}	0.87^{d}
Conophor Plantain	62.8^{d}	57^{d}	82.94^{a}	28.48^{e}	0.23^{d}	0.76^{e}
Melon Plantain	84.2^{a}	85.03^{a}	51.04^{c}	109.84^{a}	0.88^{a}	2.92^{a}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Plantain - Plantain fried in African Oil Bean seed oil; Soybean Plantain - Plantain fried in Soybean oil; Conophor Plantain - Plantain fried in Conophor nut oil; Melon Plantain - Plantain fried in Melon seed oil

Table V - Proximate composition of Potato and its fried products

Samples	Ash Content	Moisture Content	Fat Content	Crude Fibre Content	Crude Protein Content	Carbohydrate Content
Potato	2.32 ± 00.40^{a}	57.58 ± 0.17^{a}	3.45 ± 0.30^{e}	15.55 ± 0.09^{c}	1.50 ± 0.03^{b}	19.42 ± 1.33^{d}
AOB Potato	0.65 ± 0.02^{d}	31.80 ± 0.16^{c}	26.72 ± 0.70^{d}	14.68 ± 0.13^{d}	2.21 ± 0.12^{a}	23.94 ± 0.55^{c}
Soybean Potato	0.45 ± 0.01^{e}	29.21 ± 0.04^{d}	28.66 ± 0.38^{c}	15.88 ± 0.83^{b}	1.49 ± 0.02^{b}	24.31 ± 1.18^{b}
Conophor Potato	0.85 ± 0.03^{c}	41.56 ± 0.05^{b}	31.99 ± 0.10^{b}	16.43 ± 0.11^{a}	2.21 ± 0.05^{a}	6.96 ± 0.24^{e}
Melon Potato	1.44 ± 0.13^{b}	28.00 ± 0.99^{e}	35.03 ± 0.90^{a}	8.95 ± 0.15^{e}	1.50 ± 0.10^{b}	25.06 ± 0.21^{a}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Potato - Potato fried in African Oil Bean seed oil; Soybean Potato - Potato fried in Soybean oil; Conophor Potato - Potato fried in Conophor nut oil; Melon Potato - Potato fried in Melon seed oil

Table VI - Mineral content of potato and its fried products

Samples	Na	K	Ca	Mg	Zn	Fe
Potato	89.10^{c}	101.03^{a}	31.90^{d}	24.41^{c}	0.19^{bc}	9.65^{a}
AOB Potato	90.33^{b}	100.83^{b}	44.66^{e}	25.38^{b}	0.20^{b}	0.53^{c}
Soybean Potato	87.25^{d}	86.88^{e}	57.12^{b}	13.18^{d}	0.09^{d}	0.33^{d}
Conophor Potato	79.13^{e}	98.12^{d}	57.42^{a}	12.20^{e}	0.10^{cd}	0.31^{d}
Melon Potato	103.00^{a}	99.20^{c}	57.03^{b}	101.70^{a}	0.94^{a}	2.71^{b}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Potato - Potato fried in African Oil Bean seed oil; Soybean Potato - Potato fried in Soybean oil; Conophor Potato - Potato fried in Conophor nut oil; Melon Potato - Potato fried in Melon seed oil

Table VII - Proximate composition of Yam and its fried products

Samples	Ash Content	Moisture Content	Fat Content	Crude Fibre Content	Crude Protein Content	Carbohydrate Content
Yam	0.86 ± 0.03^{e}	55.56 ± 0.24^{a}	0.50 ± 0.30^{e}	16.92 ± 0.10^{c}	2.95 ± 0.18^{b}	23.20 ± 0.09^{a}
AOB Yam	1.74 ± 0.12^{b}	25.83 ± 0.19^{e}	36.91 ± 0.34^{c}	19.19 ± 0.28^{b}	263 ± 0.15^{d}	13.70 ± 0.10^{d}
Soybean Yam	1.21 ± 0.04^{d}	37.51 ± 0.23^{b}	17.78 ± 0.22^{d}	21.91 ± 0.73^{a}	2.77 ± 0.02^{c}	18.82 ± 0.74^{b}
Conophor Yam	1.36 ± 0.11^{c}	32.41 ± 0.07^{c}	37.17 ± 0.27^{b}	12.26 ± 0.08^{d}	3.60 ± 0.37^{a}	13.20 ± 0.14^{e}
Melon Yam	1.90 ± 0.08^{a}	30.38 ± 0.30^{d}	38.31 ± 0.17^{a}	11.73 ± 0.19^{e}	2.28 ± 0.06^{e}	15.40 ± 0.18^{c}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Yam - Yam fried in African Oil Bean seed oil; Soybean Yam - Yam fried in Soybean oil; Conophor Yam - Yam fried in Conophor nut oil; Melon Yam - Yam fried in Melon seed oil

Table VIII - Mineral content of Yam and its fried products

Samples	Na	K	Ca	Mg	Zn	Fe
Yam	30.8^{e}	32.12^{b}	19.14^{e}	28.17^{b}	0.24^{a}	0.76^{a}
AOB Yam	35.6^{a}	30.82^{d}	22.33^{d}	22.37^{d}	0.16^{ab}	0.35^{c}
Soybean Yam	34.75^{b}	31.2^{c}	24.24^{c}	28.03^{c}	0.2^{ab}	0.71^{a}
Conophor Yam	34.17^{c}	31.18^{c}	44.68^{b}	40.68^{a}	0.11^{b}	0.42^{bc}
Melon Yam	33.1^{d}	33.8^{a}	57.00^{a}	18.31^{e}	0.15^{ab}	0.49^{b}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Yam - Yam fried in African Oil Bean seed oil; Soybean Yam - Yam fried in Soybean oil; Conophor Yam - Yam fried in Conophor nut oil; Melon Yam - Yam fried in Melon seed oil

Table IX - Sensory evaluation of potato fried products

Samples	Taste	Aroma	Colour	Texture	General acceptability
Soybean Potato	4.50 ± 0.62^{c}	4.06 ± 0.85^{d}	7.25 ± 0.82^{a}	8.18 ± 0.43^{a}	6.00 ± 0.77^{b}
Melon Potato	8.19 ± 0.71^{a}	7.31 ± 1.02^{a}	7.38 ± 0.51^{a}	7.31 ± 1.74^{b}	7.50 ± 0.92^{a}
Conophor Potato	3.00 ± 0.84^{d}	5.44 ± 1,35^{b}	2.31 ± 0.42^{c}	4.44 ± 0.78^{d}	3.80 ± 0.64^{d}
AOB Potato	5.75 ± 0.91^{b}	5.34 ± 0.28^{c}	6.25 ± 0.12^{b}	5.88 ± 1.22^{c}	5.81 ± 1.47^{c}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Potato - Potato fried in African Oil Bean seed oil; Soybean Potato - Potato fried in Soybean oil; Conophor Potato - Potato fried in Conophor nut oil; Melon Potato - Potato fried in Melon seed oil

Table X - Sensory evaluation of plantain fried products

Samples	Taste	Aroma	Colour	Texture	General Acceptability
Soybean Plantain	5.75 ± 0.36^{c}	5.00 ± 0.64^{d}	8.31 ± 1.12^{b}	8.74 ± 0.91^{a}	6.95 ± 0.43^{b}
Melon Plantain	6.50 ± 0.72^{a}	8.06 ± 1.01^{a}	8.41 ± 0.87^{a}	7.31 ± 0.73^{b}	7.57 ± 1.32^{a}
Conophor Plantain	4.19 ± 0.51^{d}	5.21 ± 0.37^{c}	3.23 ± 0.55^{c}	4.18 ± 0.23^{d}	4.20 ± 0.52^{d}
AOB Plantain	6.00 ± 0.83^{b}	6.36 ± 0.61^{b}	8.25 ± 0.48^{b}	85.24 ± 0.59^{c}	6.46 ± 0.67^{c}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Plantain - Plantain fried in African Oil Bean seed oil; Soybean Plantain - Plantain fried in Soybean oil; Conophor Plantain - Plantain fried in Conophor nut oil; Melon Plantain - Plantain fried in Melon seed oil

obtained by Adepoju [2] where ash content was reduced in the fried product. Protein content reduced in all but conophor nut oil fried yams (Table VII). Protein content is lowered slightly albeit significantly during frying when there is high carbohydrate content though digestibility is not affected [5]. There is a formation of amylase lipid complexes during frying reducing the amount of digestible starch while increasing fiber content [12]. From Table VIII it can be seen that calcium content increased in all the yam-fried products. This is in contrast with the findings of Adepoju [2] where calcium content decreased in fried yams. Calcium plays a key role in skeletal mineralization, as well as a wide range of biological functions [24]. Iron is an essential component of hemoglobin and myoglobin. Iron content decreased in fried yams. Low levels of iron may cause Iron Deficiency Anemia [9].

In the sensory evaluation of the fried products, conophor oil fried potatoes were least preferred for taste (Tables IX, X and XI). This could be owed to undercooking as the oil was not able to penetrate into the food and expel moisture because of hardening [14]. The melon oil fried potatoes were well cooked and as a result had the best ratings for taste. The conophor nut oil fried potatoes were charred because of flames from the oil. This was probably why they were

Table XI - Sensory evaluation of yam fried products

Samples	Taste	Aroma	Colour	Texture	General Acceptability
Soybean Yam	3.25 ± 0.12^{d}	3.73 ± 0.41^{d}	6.21 ± 0.58^{b}	7.00 ± 0.42^{a}	5.05 ± 1.43^{b}
Melon Yam	7.45 ± 0.73^{a}	6.27 ± 0.52^{a}	6.47 ± 1.43^{a}	6.20 ± 0.23^{b}	6.60 ± 0.57^{a}
Conophor Yam	3.61 ± 1.04^{c}	4.32 ± 0.18^{c}	2.55 ± 0.55^{d}	3.21 ± 0.57^{d}	3.42 ± 0.82^{d}
AOB Yam	4.48 ± 0.15^{b}	4.61 ± 0.63^{b}	5.00 ± 0.61^{c}	4.53 ± 0.58^{c}	4.66 ± 0.73^{c}

*Values with the same letter superscripts in the same column are not significantly different at $p < 0.05$
Legend: AOB Yam - Yam fried in African Oil Bean seed oil; Soybean Yam - Yam fried in Soybean oil; Conophor Yam - Yam fried in Conophor nut oil; Melon Yam - Yam fried in Melon seed oil

least appreciated for color. The color of melon seed oil and soybean oil fried potatoes were best rated as they were the golden brownish color associated with properly fried foods [12]. Melon seed oil fried potatoes were generally accepted as best having the most pleasing organoleptic properties. The ratings for taste of plantain-fried products were in agreement with those given for the potato-fried products. Melon seed oil fried plantain was scored highest for aroma. The fact that the food was well cooked helped towards the development of lipid degradation products which made the aroma very appealing [12]. General acceptability ratings showed the melon seed oil fried plantain was the most accepted. The results of the sensory evaluation of yam-fried products are in agreement with the sensory evaluation of the other fried food materials. The melon seed oil fried plantain had the highest ratings for taste, aroma and color. The reasons for this has been earlier stated. Soybean oil fried plantains had the most unpleasant hydrocarbon odor that might be as a result of residual extraction solvent. The conophor nut oil fried yams had the least ratings for general acceptability.

Considering rancidity properties of the fried products at the second week of storage as shown in Table XII, the observed results are in agreement with the physiochemical properties of the oils. Conophor nut oil fried products had the highest acid values for all fried products indicating high level of hydrolytic rancidity. Peroxide values also show Conophor nut oil fried products to be most susceptible to auto oxidation. However, with the exception of plantain fried in

Table XII - Rancidity properties of fried products at week 2

Samples	Acid Value	Iodine Value	Saponification Value	Peroxide Value	Free Fatty Acids
AOB Potato	1.67	22.29	288.79	1.29	0.83
Soybean Potato	1.09	22.21	284.67	0.59	0.54
Conophor Potato	9.46	23.38	292.76	9.44	4.73
Melon Potato	1.25	26.32	290.99	0.73	0.62
AOB Plantain	1.09	26.24	259.23	25.04	0.95
Soybean Plantain	0.82	23.21	284.67	11.57	0.41
Conophor Plantain	24.47	28.22	308.16	35.73	12.23
Melon Plant	4.66	22.61	272.60	19.22	2.33
AOB Yam	3.46	21.47	291.66	5.24	1.73
Soybean Yam	7.37	16.43	253.66	10.00	0.68
Conophor Yam	13.65	25.38	246.10	11.96	3.49
Melon Yam	2.12	22.34	218.45	10.32	1.06

Table XIII - Rancidity properties of fried products at week 4

Samples	Acid Value	Iodine Value	Saponification Value	Peroxide Value	Free Fatty Acids
AOB Potato	1.96	23.78	216.50	10.62	0.98
Soybean Potato	1.22	29.4	235.84	10.02	0.41
Conophor Potato	13.75	24.02	287.48	13.93	6.87
Melon Potato	1.45	27.61	287.48	12.61	0.72
AOB Plantain	3.17	27.52	219.52	27.39	1.59
Soybean Plantain	1.82	22.53	227.73	22.87	0.41
Conophor Plantain	25.32	32.10	281.79	87.14	12.66
Melon Plant	5.26	26.81	231.35	23.21	2.63
AOB Yam	4.42	26.11	327.48	16.90	2.21
Soybean Yam	9.07	27.32	248.18	18.72	4.54
Conophor Yam	29.64	28.09	269.71	16.00	14.82
Melon Yam	2.43	23.75	183.48	14.00	1.22

Conophor nut oil, all fried products have a peroxide value below 15 mg/g showing they are still good for consumption [21].

Moreover, considering the rancidity properties of fried products at the fourth week of storage, acid value increased in all fried products (Table XIII). This is supported by the findings of Gulla and Waghray [13], which showed an increase in acid value of stored ready to eat extruded snacks. Saponification value decreased in the fried products. This is in agreement with the results of Boureghda *et al.* [6]. Peroxide value increased in all fried products [3] with most of them exceeding the 15 mg/g limit [21]. Free fatty acids naturally followed the trend of the acid values increasing in all fried products. This trend is also supported by the findings of Okonkwo *et al.* [19].

CONCLUSION

The results of this study have shown that Melon seed oil and African Oil Bean seed oil were found to be suitable frying mediums while Conophor nut oil was shown to be an unstable frying oil due to its poor physicochemical changes during and after the frying process. In terms of peroxide value. Melon seed oil showed the highest stability during frying. Melon seed oil showed the least increase in free fatty acids of the three oils indicating that it was resistant to the formation of FFAs. These results have demonstrated the potentials of these Seed Oils as good mediums for frying. The storage of fried products is accompanied by increased accumulation of primary and secondary oxidation products as measured by the Acid Value and Saponification Value. Products fried in Conophor nut oil had higher rates of accumulation of peroxides and free fatty acids compared to those fried in melon seed oil and African oil bean seed oil. This further proves Conophor nut oil as being the most unstable of the oils. Melon seed oil was best accepted in the Sensory evaluation of the fried products showing it confers pleasing organoleptic properties to food.

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Received March 2, 2016
Accepted May 4, 2016

ATTIVITÀ EDITORIALE
AREA OLI E GRASSI
ANNO 2016

Preparation of methil 9, 10 dihydroxystearic acid using a solid catalyst

P. Bondioli, L. Della Bella, G. Rivolta
Riv. Ital. Sostanze Grasse 93 (1), 5 - 10 (2016)

Dihydroxystearic acid (DHSA) is becoming a very interesting chemical for the preparation of a number of different derivatives, such as polymers, ester lubricants and azelaic/ pelargonic acids from renewable feedstocks. The classic preparation technology is represented by a one or two-step reaction carried out with hydrogen peroxyde via a peroxyacid such as performic or peracetic acid and catalyzed by a strong mineral acid. The reaction was carried out using methyl oleate as a starting material in order to avoid the formation of estolides. From oleic acid methylester an oxirane derivative on double bond is prepared and finally hydrolized to produce MeDHSA. This reaction is classically carried out using an homogenuous catalyst. During the preparation of epoxymethyl oleate catalyzed by an ion exchange resin using the in situ process via H_2O_2/peracetic acid it is possible, by using a catalyst with the same properties but different cross-linking characteristics, to drive the reaction towards a solid product that was identified as MeDHSA. The reason for this unusual behaviour stands in a different cross linkage of the ion exchange resin used. When using a resin with a low cross-linking level, the active internal acidic moieties are available for small molecules as acetic/peracetic acid as well as bigger molecules such as epoxyoleate. In this way, the hydrolisis for oxyrane moiety to MeDHSA takes place. In this paper, the main reaction conditions along with some kinetic experiments are reported and discussed.

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Nota tecnica. Lubrificanti - Corrispondenze tra metodi analitici (gennaio-dicembre 2015)

M. Sala, F. Taormina, R. Maina, P. Ruggieri
Riv. Ital. Sostanze Grasse 93 (1), 53 - 64 (2016)

Da diversi anni viene pubblicata una guida, a disposizione di chi lavora nel settore dei lubrificanti, in cui sono riportati i controlli maggiormente utilizzati per la caratterizzazione dei prodotti petroliferi e lubrificanti e i relativi metodi di analisi pubblicati da Enti Nazionali ed Internazionali (UNI, CEI, ASTM, IP, ISO, IEC, EN).

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Sulla determinazione dei tocoferoli, tocomonoenoli, tocodienoli, tocotrienoli e loro esteri negli oli vegetali

A. Gasporoli, C. Mariani
Riv. Ital. Sostanze Grasse 93 (2), 77 - 94 (2016)

Usually, the term vitamin E indicates a group of compounds that have vitaminic and antioxidants properties, known as Tococromanols. The most widely known in this group of substances are the tocopherols and tocotrienols.

Since the mid-90s, also the monounsaturated derivatives Tocomonoenols were reported in several vegetable oils, as well as Tocodienols, diinsatured derivatives of Tococromanols. The study investigated the tococromanol composition of some special oils used not only in food, but also in cosmetics. This is the case of Argan, Kukui, Sacha Inchi, and Prickly Pear oil.

It is definitely worth noting that Tocomonoenol is quantitatively the second Tococromanol in oils in which there is only one Tocopherol majority (> 90%), as in the case of Extra Virgin Olive Oil, Palm, Sunflower, Safflower, Kukui, etc.

Alongside these forms, we also highlighted ester derivatives of tocopherols and tocotrienols, primarily in palm oil, but small amounts were also found in olive oil.

It was interesting to identify of Tocomonoenolics and Tocodienolics forms, that are isomeric between them.

In refined palm oil were found different isomers of tocotrienols, and such isomerism is probably due to the action of bleaching earth on the double bonds present in the alkyl chain.

Besides the nutritional aspect, it is interesting to observe how Tococromanols esters can be used as markers to detect possible fraud since, due to their molecular size, they are unlikely to be removed during the refining process

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On the complexity of sterol fraction in olive oil

C. Mariani
Riv. Ital. Sostanze Grasse 93 (3), 147 - 155 (2016)

The sterol fraction of olive oil is much more complex than shown in the composition tables of the various legislations. In particular, lampant oils and those from Pomace show the presence of significant amounts of Ergosterol, typical sterols of fungi and yeasts.

The presence of Ergosterol appears as a large asymmetric peak eluted before Campesterol, the asymmetry is probably due to its high unsaturation that can cause degradation phenomena and adsorption favored by temperature.

Along with Ergosterol there are a number of sterols that, if not carefully monitored, can create problems

on the allocation of authenticity.
In particular, there are sterols that elute with retention times of Brassicasterol that can simulate the presence of oil of Rapeseed especially in the light of decision trees that predict the increase in Campesterol up to 4.8%. Another problem are the sterols coeluting with, or shortly after, stigmasterol that can lower the value of Sitosterol apparent.
Another problem is that with coeluiting Stigmasterol may exceed the value of Campesterol, which is not permitted by legislation.
Lastly, these sterols can erroneously simulate the presence of Δ7 Campesterol above the legal limit simulating the presence of sunflower oil.
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Technical Note. Evaluation of products for hard surface cleaning on a mineral greasy soil using colorimetric measurements
D. Mariani
Riv. Ital. Sostanze Grasse 93 (3), 173 - 174 (2016)
Aim of the test - To evaluate the efficiency in the removal of a mineral greasy soil of some trigger products for hard surfaces cleaning used with a direct contact way of action with also different contact times.
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Olio di semi di canapa: Composizione chimica e tecnologie di produzione
P. Bondioli
Comunicazione presentata al convegno *"La filiera della Canapa e il CNR",* Roma 21 Ottobre 2016
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Nuovi materiali e nuove tecnologie per la preparazione di biodiesel.
P. Bondioli
Comunicazione presentata al convegno *"19° giornata UNICHIM – Prova Interlaboratorio Prodotti Petroliferi".*
Milano, 8 Novembre 2016
La presentazione è scaricabile al seguente indirizzo:
http://www.innovhub-ssi.it/web/stazione-sperimentale-per-i-combustibili/riunioni-plenarie-2016

Sulla presenza anomala di eritrodiolo negli oli extra vergini di oliva della varietà Verdial
C. Mariani
Riv. Ital. Sostanze Grasse 93 (4), 211 - 218 (2016)
The unusual presence of erythrodiol in Verdial variety extra virgin olive oils For a long time the chemical composition of olive oils has remained unchanged, even though, the advent of globalization, has brought about a there is an awareness, in the last decade of oils that, even if genuine, possess a parameter that exceeded the limits.
For this reason, Italian researchers propose to the international community the creation of decision trees that permitted, within certain limits, a compositional anomaly in the presence of suitable guarantee standards. The anomalies best known are those of the Δ7 stigmastenol for some Syrian productions, Turkish and Greek in most of the cases, for the Campesterol exceeding the 4% limit mainly for the Arbequina and Barnea varieties, especially those produced in the Southern Hemisphere.
There are other compositional anomalies but they involve lower volumes, one of these, mainly concentrated in Spain and Portugal is linked to the presence of Erythrodiol in quantities exceeding the legal limits.
The Erythrodiol is present in all classes of olive oils; in particular, it is rich in pomace oil, 5 to 10 times compared to that contained in the olive oil obtained by physical extraction.
The amount of discovery of Erythrodiol exceeding the legal limits might suggest being in the presence of extraction oil, although the oil under examination could actually be an Extra virgin.
To clarify the situation there are various possibilities, one linked to the absolute content of erythrodiol, the other brought to light a possible marker that allows distinguishing these situations, identified in this work Erythrodiol monoesterified.
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CONGRESSI

2017 International Fuel Ethanol Workshop & Expo
19 - 21 June, 2017
Minneapolis Convention Center - Minneapolis, Minnesota
From its inception, the mission of the event has remained constant: the FEW delivers timely presentations with a strong focus on commercial-scale ethanol production – from quality control and yield maximization to regulatory compliance and fiscal management. The FEW is also the ethanol industry's premier forum for unveiling new technologies and research findings. The program extensively covers cellulosic ethanol while remaining committed to optimizing existing grain ethanol operations
Further updates on:
http://www.fuelethanolworkshop.com/ema/DisplayPage.aspx?pageId=Home_More

2017 National Advanced Biofuels Conference & Expo

19 - 21 June, 2017

Minneapolis Convention Center - Minneapolis, Minnesota

With a vertically integrated program and audience, the National Advanced Biofuels Conference & Expo is tailored for industry professionals engaged in producing, developing and deploying advanced biofuels including cellulosic ethanol, biobased platform chemicals, polymers and other renewable molecules that have the potential to meet or exceed the performance of petroleum-derived products.

Further updates on:

http://advancedbiofuelsconference.com/ema/DisplayPage.aspx?pageId=Home

8th European Symposium on Plant Lipids

2 - 5 July 2017

Malmö, Sweden - [updated 08.07.2016]

The 8th European Symposium on Plant Lipids will take in the City of Malmö 2-5th July 2017.

The conference will take place at Scandic Hotel in the city center of Malmö.

Apart from covering most recent achievements in both basic and applied Plant Lipid Science the conference will also cover emerging topics related to lipids that have not been much covered in previous meetings, such as the role of lipids in plant autophagy. Apart from invited speakers introducing the different sessions, short talks will be selected from submitted abstracts in each session. There will also be a poster session.

SCIENTIFIC PROGRAMME [updated 01.11.2016]:

Sunday, 2 July 2017

16:00 Registration

17:00-20:00 Welcome reception

Monday, 3 July 2017

08:15-08:30 Welcome

- Session 1: Extracellular lipids
  08:30-09:00 Keynote lecture:
  New insights into biosynthesis of cuticular wax. Ljerka Kunst (University of British Columbia, Canada)
  09:00-10:00 Selected oral presentations
  10:00-10:30 Coffee break
- Session 2: Sphingolipids and sterols
  10:30-11:00 Keynote lecture:
  Sphingolipids: new players in plant defense. Sandrine Dhondt-Cordelier (The University of Reims Champagne-Ardenne, France)
  11:00-12:00 Selected oral presentations
  12:00-13:00 Lunch
- Session 3: Lipids and environmental interactions
  13:00-13:30 Keynote lecture:
  Acylethanolamide signaling supports seedling survival under environmental stress. Kent Chapman (University of North Texas, USA)
  13:30-14:30 Selected oral presentations
  14:30-15:00 Coffee break
- Session 4: Lipid membrane dynamics
  15:00-15:30 Keynote lecture:
  Flippases - lipid transporters in cellular membranes
  Thomas Günther-Pomorski (Copenhagen University/ Ruhr University Bochum, Germany)
  15:30-16:30 Selected oral presentations
  16:30-18:00 Poster session

Tuesday, 4 July 2017

- Session 5: Storage lipid biosynthesis and metabolism
  08:30-09:00 Keynote lecture:
  Storage lipid metabolism in the model green microalga Chlamydomonas reinhardtii. Yonghua Li-Beisson (Biosciences and Biotechnologies Institute of Aix-Marseille, France)
  09:00-10:00 Selected oral presentations
  10:00-10:30 Coffee break
- Session 6: Lipid catabolism and recycling
  10:30-11:00 Keynote lecture: *TBA*
  11:00-12:00 Selected oral presentations
  12:00-13:00 Lunch
- Session 7:Lipid metabolic modelling and flux analysis
  13:00-13:30 Keynote lecture:
  Using flux analysis to help understanding of lipid metabolism. John Harwood (Cardiff University, UK)
  13:30-14:30 Selected oral presentations
  14:30-15:00 Coffee break
  15:00-17:00 Poster session
  18:00-23:00 Complimentary Conference Dinner hosted by Malmö City, Malmö Town Hall.

Wednesday, 5 July 2017

- Session 8: Lipid biotechnology
  08:30-09:00 Keynote lecture:
  EPA/DHA plant oils arrive – three decades of omega-3 research from inspiration to innovation. Allan Green (CSIRO Agriculture & Food, Australia).
  09:00-10:00 Selected oral presentations
  10:00-10:30 Coffee break
  10:30-11:00 Discussions of contents and location of next ESPL conference
  13:00-14:00 Post conference tour - Cultural/City tour (optional)

For updates and information:

http://www.eurofedlipid.org/meetings/malmoe2017/index.php

Email: info@eurofedlipid.org

2017 EFITA CONGRESS

2-6 July 2017

Montpellier, France

EFITA is the European conference dedicated to the future use of ICT in the agri-food sector, bioresource and biomass sector. It was launched and is supported by the European Federation for Information Technology in Agriculture, Food and the Environment (EFITA).

The conference will be held ah the Montpellier Supagro school, Montpellier, France, on July 2-6, 2017 and its official language will be English.

Conference Main Topics

The topics for the EFITA 2017 conference are detailed below within topic groups. Topics of invited sessions and associated workshops will be soon announced.

• *Topic group "ICT for farming":*

This topic group is meant to invite communications about design of computer tools for farming, models of farming activity, scientific computing applied to crop management, precision agriculture, expert systems. It includes topics such as

- ICT applications for agriculture and sustainability
- ICT applications for precision farming and knowledge intensive agriculture
- Decision Support Systems for Agriculture

• *Topic group "The web, the field, the farm, the business":*

This topic group is meant to invite communications about web technologies and networking of actors all along the value chain of agriculture. It includes topics such as

- On line farm services
- Web applications (clients, devices, server-side)
- Cloud computing applications
- Social Networking, collaborative tools and crowdsourcing
- Tools for e-agribusiness

• *Topic group "Remote sensing and planning":*

This topic group is meant to invite communications about remote sensing, GIS technologies and spatial management of resources. It includes topics such as

- Remote Sensing and GIS applications
- Planning tools
- Environmental information systems and Environmental management systems
- ICT applications for natural resources management, including forestry
- ICT applications for sustainable biomass production and use

• *Topic group "Sensing, robotics and electronics for agriculture":*

This topic group is meant to invite communications about signal processing, sensing technologies, automation and control sciences, and embedded computing, for agriculture. It includes topics such as

- Sensing – Image Processing
- Robotics in Agriculture and machine embedded ICT tools
- Internet of Things (IoT) in the field and in the supply chain
- Wireless sensor networks

• *Topic group "Simulation and models for agriculture":*

This topic group is meant to invite communications about agronomy and ICT, with a focus on modeling for simulation, prediction, crop management, design of ICT-intensive farming systems. It includes topics such as

- Modeling and Simulation for agricultural production and farming systems
- Weather prediction models for sustainable agricultural production
- Multi-Agent systems

• *Topic group "The food chain and agricultural ICT policies":*

This topic group is meant to invite communications about economical and business implications of ICT in agriculture, as well as about the design of ICT tools that meet organizational needs. It includes topics such as

- ICT applications for food chain and logistics
- Traceability tools
- ICT and business
- Rural economies and ICT policies for rural development (northern and southern countries)

• *Topic group "Semantic interoperability and Knowledge Management":*

This topic group is meant to invite communications related to Information System design for agriculture and natural resources management, with a focus on interoperability, semantics and knowledge management. It includes topics such as

- Metadata and data standards in agriculture
- Thesaurus management, Knowledge management
- Ontologies for agriculture
- Knowledge bases and Knowledge repository services
- Web of Data, Linked Open Data

• *Topic group "Big data, analytics and visualization":*

This topic group is meant to invite communications related to Information Systems and algorithmics for agriculture and natural resources management, with focus on data mining, data warehousing, visualisation, knowledge extraction, big data management. It includes topics such as

- Big data and data mining for agricultural information systems
- Data visualisation
- Management of data and knowledge, incl. case studies (for example in extension and advice services).
- ICT for farming
- The web, the field, the farm, the business
- Remote sensing and planning
- Sensing, robotics and electronics for agriculture
- Simulation and models for agriculture
- The food chain and agricultural ICT policies
- Semantic interoperability and Knowledge Management
- Big data, analytics and visualization

For update and information:
http://www.efita2017.org/

ECPA - 11th European Conference on Precision Agriculture

16 - 20 July 2017

Edinburgh Scotland

It is 20 years since the first ECPA conference and the UK organisers are pleased to welcome the return of the conference to the UK and to Edinburgh. The conference will continue with a successful format of previous conferences building in strong industry sessions and participation. The theme of 'Innovating through Research' will enable all involved in Precision Agriculture to participate. Oral and poster presentations will be welcomed from authors on any precision agriculture topic though particularly welcome in the list of topics shown in the Programme section. All prospective authors and presenters should view the 'Key Dates' section to ensure they can meet the deadlines.

Registration is for ECPA 2017 is not yet open, but anyone can record their interest and receive regular updates by pre-registering.

Edinburgh is Scotland's capital city with unique history and architecture. It's a major UK centre of agricultural and land-based research where there are leading centres for precision agriculture and sensor-based technology with strong industry links.

On ECPA's 20th anniversary, Edinburgh will host the first return to the UK since the first Conference and provides a new and exciting venue for ECPA. In addition to a strong academic programme, the conference will feature strong links to industry with practical input through commercial participants and the UK's new Centres of Excellence in Innovation.

The conference will be based at the University of Edinburgh's John McIntyre Conference Centre, within the Pollock Halls Centre.

Delegates will need to register for different elements of the programme.

If you have any queries, please email:
info@ecpa2017.com

Further updates on:
https://ecpa.delegate-everything.co.uk/

15th Euro Fed Lipid Congress

27 - 30 August, 2017

Uppsala, Sweden - hosted by Nordic Lipid forum [updated 15.02.2017]

Dear Colleagues, Nordic Lipidforum has the great honor to host the 15th Euro Fed Lipid Congress 27-30 August 2017 in Uppsala, Sweden.

The symposium will take place at the Uppsala Concert & Congress (UKK) located in the very city center of Uppsala, within short walking distance from the train station, all hotels and the historical center of Uppsala.

The theme of the symposium reflects the great importance of oils, fats and lipids for a healthy life through its multiple functions in all aspects of human life and care. The Scientific committee and the Nordic Lipidforum warmly welcome all scientists with an interest in lipids to join the Euro Fed Lipid Congress in Uppsala in August 2017.

Main Topics/ Keynote Lectures:

- *Analytics/Lipidomics:*
  Christer Ejsing, University of Southern Denmark, Odense, Denmark: "Shotgun Lipidomics: Harnessing the Power of High Resolution Mass Spectrometry for the Analysis of Complex Lipid Samples"
- *Biotechnology and Enzyme Technology:*
  Marc Kellens, DeSmet Ballestra Group, Zaventem, Belgium: "Implementation of Biotechnology Solutions on Oils and Fats Processing"
- *Animal Lipids, including Dairy:*
  Laurence Bernard, INRA, France: "Advances in Nutritional Strategies to alter the Lipid Fraction of Ruminant Milk for a Healthier Life"
- *Health and Nutrition:*
  Berthold V. Koletzko, Univ. of Munich Medical Center, Germany: "Early Life PUFA Intake Interacts with Genetic Variation in Determining Health Outcomes" Lotte Lauritzen, University of Copenhagen, Denmark: Effects on Cognitive Development by Maternal and Infant LC n-3 PUFA Intake"
- *Plant Lipids and Plant Breeding:*
  Sten Stymne, SLU, Alnarp, Sweden: "Three Decades of Plant Oil Biotechnology: Where are we today and how do we move forward?"
- *Oleochemistry, Biofuel and Biosurfactants:*
  Jean-Luc Dubois, Arkema, Colombes, France: "Preparation of Monomers from Fatty Acids through various Chemistries"
- *Seed Oils and Speciality Oils – including Rapeseed, Berry and Olive Oil:*
  Parkash Kochhar, Reading, UK: "Emerging Importance of Speciality Oils: Characteristics and Beneficial Aspects"
- *Palm Oil:*
  Ahmad Kushairi Din, Kuala Lumpur, Malaysia: "Mitigation Actions by the Malaysian Palm Oil Industry to reduce the formation of 3-MCPD and GE in Palm Oil"
- *Lipid Oxidation and Quality:*
  (supported by Kalsec) Giuseppe Poli University of Torino, Italy: "Lipid Oxidation Products between Health and Disease"
- *Marine Lipids:*
  Bente Ruyter, Norwegian Institute of Food, Fisheries and Aquaculture Research, Aas, Norway: "Strategies to improve Efficiency of Omega-3 Fatty Acid Utilization in Atlantic Salmon"
- *Physical Chemistry:*
  Anna Fureby, SP Technical Research Institute of Sweden, Stockholm, Sweden: "Superficial Investigations - Characterisation Techniques for Surface related Phenomena in Lipid Containing Systems"
- *Processing and Sustainable Sourcing:*
  Karsten Nielsen Arahus Karlshamn AB, Malmö,

Sweden: "Process & Sourcing Challenges - a Historical and Future Perspective"
- *Microbial and Algal Lipids:*
  Jean-Marc Nicaud, INRA, Thiverval Grignon, France: "Engineering of Lipid Metabolism in Yarrowia lipolytica"
- *Lipids in Formulation of Pharmaceutics and Cosmetics:*
  Christel Bergström Department of Pharmacy, Uppsala University, Sweden: "Can Dietary Lipids Enable Delivery of Problematic Pharmaceutical Compounds?"
- *Lipids in Novel Foods:*
  Marina Heinonen, University of Helsinki, Finland: "Lipid Containing Novel Foods and their Safety"
- *Lipid Soluble Contaminants:*
  Jacob de Boer, VU Environment and Health, Free University Amsterdam, The Netherlands: "Contaminants in Lipids: An Undesirable yet Growing Group of Chemicals"

Organiser:
European Federation for the Science and Technology of Lipids e.V.
Postfach 90 04 40
60444 Frankfurt/Main
Phone: +49 69/79 17-533
Fax: +49 69/79 17-564
E-Mail: info@eurofedlipid.org
Please mark your calendars!
Further updates on:
http://www.eurofedlipid.org/meetings/uppsala2017/index.php

2017 Cannabis Science Conference

August 28-30, 2017
Portland Oregon USA

Cannabis Science Conference is the world's largest cannabis science expo. Our conference pulls together cannabis industry experts, Instrument manufacturers, testing labs, research scientists, medical practitioners, policy makers and interested novices. Our annual event is aimed at improving cannabis science. Join us in Portland, Oregon, for an exciting conference with keynotes, presentations, round table discussions and exhibits. At our inaugural event we hosted over 750 attendees from all over the world!
Grow with us in 2017 at the Oregon Convention Center in Downtown Portland, OR, August 28th-30th! This year we will have 60,000 square feet of exhibit hall space and 50,000 square feet of meeting space for technical presentations. We hope that you will join us for this historic event.
Don't miss out on our 2017 Canna Boot Camp! This is our full-day workshop that covers everything from Cultivation, Extraction, Sample Prep, Analytical Testing, Edibles Manufacturing and more! This event sold out very quickly in 2016.
- 60,000 sq. ft. of exhibit hall space
- 50,000 sq. ft. of meeting space for presentations
- Parallel medical & scientific technical sessions
- Expanded Canna Boot Camp with new cultivation segment
- Technical poster presentations and much, much more!

For update and informations:
https://www.cannabisscienceconference.com/

4th High Oleic Oils Congress (HOC2017)

September 5-6, 2017
Bucharest - Romania

Dear Colleagues, The 4th High Oleic Oils International Congress will occur from 5 to 6 September 2017 in Bucharest, Romania.
Why Romania? During the past 5 years, Romania experimented one of the fastest expansion of HO sunflower acreage, at 39% annual growth rate.
Hence, Romania became a leading country in Central Europe for HO sunflower trading, at crossroads between Asia and Europe. Romania also experimented the cultivation of HOLL rapeseed. How far both HO crops will expand in the country and in Central Europe? We will surely address this question.
The HOC congress is now turning global, becoming the meeting place for leaders in the HO value chain. More than 250 producers, traders and buyers are expected to come from 40 different countries.
The new season is recovering from the past year surpluses, and demand will require an increase in acreage and oil production. What would be the engines of growth: The reaction of food companies to the new saturated fat labelling entering into effect in the european community? Chinese hunger for HO oils? The emerging GM-free trend in US? The premium price in 2017/18? The new business models linking agribusiness and oil producers?
Let's discuss in Bucharest, learn more about the HO market, and network in a splendid town built in the XVth century!
PRELIMINARY PROGRAM
- *Tuesday, September 5th*

9:00-10:00 Registration
10:00-10:30 Welcome & Congress Introduction
SESSION 1 – AGRICULTURAL LANDSCAPE & PRODUCTION ANALYIS
10:30-11:00 HO Oilseeds & Oils 2017 Market in North America – Jim Johnson (Star Specialty Seed Inc., USA)
11:00-11:20 HO Oilseeds & Oils 2017 Market in Latin America – Archibaldo Salvador (Syngenta, Argentina)
11:20-11:40 HO Oilseeds & Oils 2017 Market in the Danube Area – Cédric Delavent (Euralis, France)
11:40-12:00 HO Oilseeds & Oils 2017 Market in Ukraine and Russia – Svetlana Synkovskaya (APK Inform, Ukraine)
12:00-12:30 Development of High Oleic-high Yielding

Safflower Cultivars Suitable to Indian Conditions – Anjani Kammili (Indian Institute of Oilseeds Research, India)
12:30-13:00 Quality Monitoring and Traceability for HOLL Rapeseed Production from Field to Oil – Lionel Lordez (Monsanto, France)
13:00-14:30 Lunch
SESSION 2 – CHANGES IN HO OILS DEMAND
14:30-15:00 Panel Discussion: Will HO Oilseeds Production Meet Demand ?
15:00-15:20 Forecasting HO Oils Demand in India - Aman Jyoti (Marico Ltd, India)
15:20-15:40 Decrypting the Emerging High Oleic Oils Consumption in Brazil – Emilio Figer (Celena Alimentos, Brazil)
15:40-16:00 Changing High Oleic Oils Demand in Western Europe – Perrine Tonin (Avril Group, France)
16:00-16:30 Networking Coffee Break
16:30-17:00 High Oleic Oil Production and Usage in South Africa – Rouxlene Van Der Merwe (University of Free State, South Africa)
17:00-17:30 How much Palm Bashing is reinforcing the HO Oils Consumption in the EU? – TBC (FAT & Associés, France)
17:30-18:30 Exhibition Area

- *Wednesday, September 6th*

8:00-9:00 Exhibition Area
9:00-9:30 Positioning High Oleic Oils in Nestle's Products – Constantin Bertoli (Nestlé, Switzerland)
9:30-10:00 Artificial trans-fat in Popular Foods in Countries in Eastern Europe, Central Asia and Transcaucasia – Steen Stender (Copenhagen University, Denmark)
10:00-10:30 High Oleic Canola Oil Enriched with DHA: A Powerful Combination for Health Promotion – Celia Rodriguez Pérez (CIDAF, Spain)
10:30-11:00 Networking Coffee Break
11:00-11:30 Positioning (HO oils) Specialities in the Oilseeds Agriculture – David Jackson (LMC International, UK)
11:30-12:00 True Costs Differential in Growing High Oleic Sunflower in France – Hugues de Durand (SCEA de Maisonneuve, France)
12:00-12:30 Can Industry stabilize the Premium Prices for a long Period? – Jean Massiani (FAT & Associés, France)
12:00-12:30 The Global Crop Protection & Seed markets – short term prospects focusing on M&A in Agribusiness and its impact on the Oilseeds Agriculture – Bob Fairclough (Kleffmann, Germany)
13:00-14:30 Lunch
SESSION 3 – INNOVATION & INVESTMENT POTENTIAL
14:30-15:00 What's changing in the Global High Oleic Game? – Luis Carlos Alonso (Syngenta, Spain) & Fabrice Turon (FAT & Associés, France)
15:00-15:20 Adding Value and Improving Meal Quality in Rapeseed – Richard Burrell (Dow Seeds, UK)
15:20-15:40 Functional Gains in Vegetable oils: High Oleic Soybean Oil – Frank Flider (US Soybean Export Council, USA)
15:40-16:00 Specialty Trait Oils: Past, Present and Future Primary – Willie Loh (Cargill, USA)
16:00-16:30 Networking Coffee Break
16:30-17:30 Panel Discussion: What's Hot in the HO Oils Market?
19:30-22:30 Gala Dinner
For update and information:
http://higholeicmarket.com/hoc-2017/

World Soybean Research Conference Ten And the 17th Biennial Conference on the Molecular and Cellular Biology of Soybean

September 10-15, 2017
Savanna, Georgia, USA

Program in progress, new information coming soon. Save the date.
Email: katkinson@asginfo.net
For update and information:
http://wsrc10.net/

17th AOCS Latin American Congress and Exhibition on Fats and Oils

11 - 14 September, 2017
Grand Fiesta Americana Coral Beach Hotel - Cancun, Mexico

Enhance your knowledge of today's market

- Gain an overview of the industry now and what to expect in the future
- Experience more than 80 invited and volunteer presentations
- View poster sessions with innovative research
- Connect with colleagues during the exhibition and social events

Topics

- Analytical and Quality Control
- (Bio) Detergents, Control Cosmetics, and Surfactants
- Biodiesel
- Biotechnology: Interesterification, Bio-products, and Enzymatic Process
- By-products: Protein, Lecithin, Fiber, Tocopherols, and more
- Extraction, Refining, and Processing
- Fats in Pet Foods
- Health and Nutrition
- Oxidation of Lipids and Antioxidants in Food
- Regulatory and Commercial Aspects Affecting the Fats and Oils Industry: 3MCPD, PHA, *trans*, and more
- Sensory Evaluation in Fats
- Specialty Oils: Olive, Avocado, Algal Oil, and more
- Structure, Functionality, and Applications in Food
- Sustainability in Fat Production

Further updates on:
http://lacongress.aocs.org/

Oils & Fats International Trade Fair for Technology and Innovations

12 - 14 September, 2017

Munich, Germany

The oils + fats will take place on 3 days from Tuesday, 12 September to Thursday, 14 September 2017 in Munich.

The oils+fats is an international trade fair for business, technology and innovation in vegetable and animal oils and fats. It is the only one of its kind in Europe and includes machinery, components and tools for the production and processing of oils and fats. Especially the market for vegetable and animal oils and fats is becoming more and more important and has to rely on innovation to reduce costs and to save energy through increased competition and the increasing demands in terms of environmental aspects. Because this fair shows current trends and the latest technological innovations and developments from the raw materials and auxiliaries to processing, quality assurance, packaging and logistics, it has established itself as the most important meeting point of the industry for producers, refiners and distributors. The oils+fats will be held every two years in Munich and is only open to trade visitors of the oils and fats industry. As communication and information platform, it provides an intense experience exchange with business, market and opinion leaders, and presents practical solutions to the challenges the industry faces.

Exhibits/main sectors:

Raw materials, auxiliary materials, production and processing, processing steps, products, components, overlapping processes, engineering and consulting, logistics, quality control and assurance, research, institutions and publishers, filling and packaging technology, deep frying.

Business sectors:

Chemistry, Food Processing, Food, Beverages.

For informations and further updates on:
http://www.tradefairdates.com/oils-fats-M567/Munich.html
Email: info@oils-and-fats.com

HAICTA 2017
8th International Conference on Information & Communication Technologies in Agriculture, Food and Environment

21 - 24 September, 2017

Chania, Crete, Greece

The Conference is co-organized by the Hellenic Association for Information and Communication Technologies in Agriculture, Food and Environment (HAICTA) and the Mediterranean Agronomic Institute of Chania (MAICh) in cooperation with a number of Institutions.

- Aristotle University of Thessaloniki, Greece
- Alexander Technological Educational Institute of Thessaloniki, Greece
- University of Macedonia, Thessaloniki, Greece
- Technological Educational Institute of Western Macedonia, Greece
- Agricultural University of Athens, Greece
- Technical University of Crete, Greece

HAICTA is the Greek Branch of the European Federation for Information Technology in Agriculture (EFITA). Until now HAICTA has organized a series of seven successful international conferences.

HAICTA 2017 aims to bring together professionals, experts and researchers working on Information and Communication Technologies in Agriculture, Food and Environment. We additionally aim to emphasize on the applicability of ICT solutions to real industry cases and the respective challenges.

Topics

The topics cover all sectors all areas of ICT in Agriculture, Food and the Environment including but not limited to:

- Information Systems
- Web Applications
- Database Systems and Data Mining
- Decision Support Systems
- Innovations in Food Hygiene and the Production of High Quality Food
- Quality Assurance and Certification of Innovative Food Products
- E-Business, E-Commerce, E-Sales, E-Marketing and E-Services
- Innovative Uses of Agricultural By-products and Waste Management
- Traceability Systems
- Innovation in Short Value Chains in Rural Areas
- Modeling and Simulation in Climate Change
- Water Resources Management
- Environmental Design and Policy
- Environmental Impact Assessment
- Epidemics Modelling
- Internet of Things, Sensor, RFID and Mobile/Wireless Network Applications
- Precision Farming Systems, Variable Rate Technologies
- Farm and Animal Health Monitoring and Data Recording Systems
- Efficient Irrigation Technologies and Water Quality Monitoring
- Fish Health and Product Quality Monitoring in Aquaculture
- Genomics and Biotechnologies in Genetic Improvement
- Diffusion of Innovation in Rural Areas
- E-networking, Collective Actions and E-governance in Rural Areas
- E-learning, Interactive Systems and Web-based Farmer Education
- ICT and Innovation in the Promotion of Rural Areas and Alternative Tourism
- Wood Technology and Wood Products
- Wildfire Risk Assessment

- Information Systems and Wildlife Management & Protection
- Spatial Analysis, Landscape Planning and GIS-based Analysis
- Supply Chain Management & Logistics

For any further information you can contact the conference secretariat at:
haicta2017@gmail.com or the program chairs.

Vegetable Oil Processing and Products of Vegetable Oil/Biodiesel

October 1-5, 2017

Bryan, Texas USA

Objectives of Short Course

Train production personnel in principles and practices of:

- New methods in vegetable oil refining and processing
- Latest methods in bleaching, neutralization, interesterification and deodorization of major vegetable oils
- Production of biofuels
- Production of non-trans fats
- Filtration and vacuum systems and much more

Who Should Attend

This short course is a must attend for anyone involved in the field of Vegetable Oil Processing and interested in the latest developments in bleaching, hydrogenation, interesterification, deodorization, production of biodiesel and non-trans fats.

- Plant Managers and Engineers
- R &D Personnel
- Sales and Marketing Personnel
- Quality Control and Quality Assurance Personnel
- Application Scientists

PROGRAM:

Sunday, October 1, 2017

4:15PM
Registration and Welcome Dinner - Hampton Inn, College Station
5:30 PM Short Course Orientation: How to Get the Most Out of a Training Program - Staff, Food Protein R&D Center
5:45 PM Dinner - Rudy's BBQ

Monday, October 2, 2017

7:40 AM Bus leaves hotel for Rudder Tower, Texas A&M
8:00 AM Basic of Fats and Oils Chemistry, Factors Affecting Crude Oil Quality - M.S. Alam
9:00 AM Vegetable Oil Feed Stocks and Renewable Diesel - M.Holtzapple
10:15 AM Break
10:30 AM Vegetable Oil Extraction : Pros and Cons - R. Clough
11:45 AM Lunch
12:45 PM Group Photo
1:00 PM Nano Neutralization '"A cutting edge refining Technology" - L. Espinosa
2:15 PM Break
2:30 PM Degumming of Crude Oils and Basics of Vegetable Oil Refining - W. Younggreen
3:30 PM Bus leaves for Riverside Campus - Combined acid Degumming and Caustic Refining Demonstration
5:00 PM Bus leaves for hotel

Tuesday, October 3, 2017

7:40 AM Bus leaves hotel for Rudder Tower, Texas A&M
8:00 AM Deoderizer design and heat recovery - L. Espinosa
9:00 AM Break
9:15 AM Centrifuges and Decanters in Vegetable Oil Processing.Design, Operation and Maintenance - T. Neuman
10:30 AM Break
10:45 AM Adsorptive Bleaching Materials and Processes - J. Bello
11:45 AM Lunch
1:00 PM Water Management Technology in the Refinary - W. Younggreen
2:00 PM Break
2:15 PM Filtration during oil processing- B. Boyd
3:15 PM Bus leaves for Riverside Campus - Adsorptive Oil Refining Demonstration
5:00 PM Bus leaves for hotel

Wednesday, October 4, 2017

7:40 AM Bus leaves for Rudder Tower , Texas A&M
8:00 AM Application of FT-NIR in Vegetable Oil Analyses- J. Hudson
9:15 AM Biodiesel Manufacture Principles and Equipment - B. McDonald
10:15 AM Break
10:30 AM Employing Lab Scale Equipment to Resolve Plant Scale Issues - F. Filippini
11:30 AM Lunch --> Graduation Lunch
1:00 PM Absorben Purification of Biodiesel - B. Cooke
2:00 PM Palm Oil Processing and Utilization: A New Direction for the Industry - J. Minal
3:00 PM Break
3:15 PM Bus leaves for Riverside Campus—Deodorization of Vegetable and Biodiesel Production Demonstration
5:00 PM Bus leaves for hotel

Thursday, October 5, 2017

7:40 AM Bus leaves for Rudder Tower
8:00 AM Formulations and Technologies of Non Trans Products - J. Satumba
9:00 AM Safety and Environmental Issues for Vegetable Oil Processing - J. Mulholland
9:45 AM Vaccum Systems: Operations and Troubleshooting - A. Fris
10:45 AM Enzymatic Degumming of Vegatable Oils. Recent Developments - M. Jung
11:45 AM Lunch
1:00 PM Potential Membrane Separation Applica-

tions in Vegetable Oil Refining - Y. J. Lee
2:00 PM Biobased Industrial Fluids & Lubricants - S. Awbrey
2:30 PM Short Course Adjourns
For update and information:
Email: msalam@tamu.edu
http://foodprotein.tamu.edu/fatsoils/scvegoil.php

9th International Symposium on Deep-Frying. Higher quality, safer products and further use

30 - 31 October, 2017
Shanghai, China [12.09.2016]

Welcome to the 9th international symposium on deep-fat frying. The international symposia on deep-fat frying organized through the DGF and Euro Fed Lipid have been held most of time in Europe and North America. This is the first time it is held in China and will be organized jointly by the Euro Fed Lipid and the Chinese Cereals and Oils Association.

There is a long tradition of deep frying in Asia's cuisine. The oldest recording of frying process was found more than 3000 year old back in the Chinese texts. In recent decades, the rapid development of China's economy has resulted in a dramatic increase in frying industry. We sincerely hope that this will be an opportunity for the experts and distinguished players in the area share their knowledge and experiences in Shanghai, China.

Furthermore, it will be a great chance for all of participants to get more understanding of the Chinese culinary culture, fast development and potential huge market.

SCIENTIFIC PROGRAMME [updated 28.09.2016]

- Monday, 30 October 2017

09:00 Opening Remarks
1st Session: Current status, legislation and safety
09:30 *History and Evolution of Deep-Fat Frying.* Richard F. Stier (Sonoma, CA/USA)
10:00 *Industrial frying in China: Current status and development trends.* Ruiyuan Wang/Xingguo Wang (CCOA/Jiangnan University, China)
10:30 Coffee Break
11:00 *Food safety and HACCP: Ensuring the Safety of Foods and Oils*. Richard F. Stier (Sonoma, CA/USA)
11:30 *Formation and Migration of Health-Risk Compounds during Deep Frying*. Qinzhe Jin (Jiannan University, China)
12:00 *Regulatory Issues throughout the World*. Bertrand Matthaeus (Max Rubner Institut, Detmold, Germany)
12:30 Lunch
2nd Session: Fundamentals and application
13:30 *Fundamentals of Deep Frying.* Felix Aladedunye (University of Lethbridge, Canada)
14:00 *Simulation of the Frying Process.* Christian Gertz (Maxfry, Hagen, Germany)
14:30 *Mechanism and Reduction of Oil Uptake in Deep-Fried Instant Noodles*. Jinfeng Qi (Jiangnan University, China)
15:00 Break
15:30 *Properties and Performance of Different Frying oils*. Yulan Liu (Henan University of Technology, China)
16:00 *The Application and Advantage of Rice Bran Oil for Deep Frying.* Yuanrong Jiang (Wilmar, Shanghai, China)
16:30 *Selecting Frying Oils: Oil Specifications and Performance*. Bertrand Matthaeus (Max Rubner Institut, Detmold, Germany)
17:00 *Futher of edibile oil in the frying industry Cargill*. Lucky Inturrisi, Cargill (Melbourne, Australia)

- Tuesday, 31 October 2017

3rd Session: Optimum frying
09:00 *The Application of Frying Oil concerning the Microstructure Change and Migration of Oil Soluble Compounds in Frying Flour Product*. Xiangyu Wang (COFCO, Bejing, China)
09:30 *Foodservice Frying: Fryer Design and Maintenance* (Speaker to be announced)
10:00 *Total Oil Management: oil use safety and efficiency*. Yan Liu (Cargill, Shanghai, China)
10:30 Break
4th Session: Analysis and Quality Control
11:00 *Quality Control during industrial frying*. Christian Gertz (Maxfry, Hagen, Germany)
11:30 *Analytical possibilities to monitor fat degradation during storage and heating*. Bertrand Matthaeus (Max Rubner Institut, Detmold, Germany)
12:00 *Standardization of Quick Tests for Total Polar Compounds in Frying Oil by Measuring Dielectric Constant.* Xu Li (Jiangnan University, China)
12:30 *Use of NIR for Evaluating Degrading Oils*. Christian Gertz (Maxfry, Hagen, Germany)
13:00 Lunch
5th Session: Antioxidants and stability
14:00 *Effect of nutural antioxiants on the frying performance*. Bertrand Matthaeus (Max Rubner Institut, Detmold, Germany)
14:30 *Structured Phenol Lipids based on Sunflower oil, their Application and Performance in Frying*
Lingyi Liu (Wuhan Polytech University, China)
15:00 *Flavor Characters Analysis of Fried Food in China.* Junmei Liang (Wilmar Shanghai, China)
15:30 Break
16:00 *Occurence and Measurement of Low Molecular Volatile Compounds in the Fume of Different Frying Oils*. Bertrand Matthaeus (Max Rubner Institut, Detmold, Germany)
16:30 *The Factors Affecting Texture and Taste of Traditional Chinese Dough Sticks*. Wenbo Mu, (Wilmar, Shanghai, China)
17:00 Final Discussion
For updates:
http://www.eurofedlipid.org/meetings/shanghai2017/

XX Lipid Meeting

December 7-9, 2017

Leipzig Germany

The XX Lipid Meeting Leipzig will be held December 07-09, 2017 at the Salles de Pologne/Events & Konferenzen, Germany.

Since its foundation more than twenty years ago, this meeting in the heart of Germany has developed into an established forum for scientific discussion and interdisciplinary collaboration in all relevant areas of lipid metabolism and diseases caused by lipid disorders.

This year's Lipid Meeting will focus on results from genetic studies of lipid metabolism and atherosclerosis, recent developments for the treatment of lipid disorders and prevention of cardiovascular disease, as well as lipid biology in adipose tissue and its relation to cardiometabolic disease. The Lipid Meeting Leipzig brings together an international group of participants from all career levels and from various scientific and medical disciplines to present cutting-edge research in a collegial atmosphere.

We hope to offer you an interesting and scientifically exciting meeting and look forward to welcoming you in Leipzig.

Prof. Dr. Joachim Thiery
Prof. Dr. Uta Ceglarek
Prof. Dr. Ralph Burkhardt
Institut für Laboratoriumsmedizin, Klinische Chemie und Molekulare Diagnostik

Main Topics

- Novel therapeutic approaches to lipid disorders and cardiovascular disease
- Lipids, obesity and diabetes
- Lipid trafficking and cellular homeostasis
- Cross-talk between lipid metabolism and immune response
- Genetics of lipid metabolism and atherosclerosis
- New aspects and pathways in atherosclerosis
- Epidemiology of lipid-mediated diseases: risk factors, prevention and novel biomarkers

For update and information:
Email: jkaftan@eventlab.org
http://www.lipidmeeting.de/

109th AOCS Annual Meeting

06 - 09 May, 2018

Minneapolis, USA

Please mark in your calendars.
For updates:
Contact: AOCS Meetings Department, USA
Telephone: 1 217 6934821
Email: meetings@aocs.org

Institute of Food Technologists Annual Meeting and Food Expo

14 - 18 July, 2018

Chicago US (United States)

IFT Food Expo is a 4 day event being held from 15th July to 18th July, 2018 at the McCormick Place - South Hall in Chicago, United States Of America. This event showcases products like spread of food products and services, food materials, raw materials and ingredients, packaging products and services, food supplying services, food processing and preparing equipment, food preparing technologies and other food related products and services, is a must attend event for all etc. in the Food & Beverage industry.

Further updates on:
http://10times.com/ift-food-expo

16th Euro Fed Lipid Congress

16 - 19 September, 2018

Belfast, UK, Hosted by SCI Lipidgroup

Please mark in your calendars
Organiser:
European Federation for the Science and Technology of Lipids e.V.
Postfach 90 04 40
60444 Frankfurt/Main
For updates:
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Fax: +49 69/79 17-564
E-Mail: info@eurofedlipid.org

Fabric and Home Care World Conference

28 - 31 October, 2018

Boca Raton, Florida, USA

Please mark in your calendars
For updates:
Email: meetings@aocs.org

INNOVHUB
STAZIONI SPERIMENTALI
PER L'INDUSTRIA

SSOG
STAZIONE SPERIMENTALE
PER LE INDUSTRIE DEGLI OLI E DEI GRASSI

Innovazione e ricerca

RISG

LA RIVISTA ITALIANA DELLE SOSTANZE GRASSE

2017

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antonella.spazian@mi.camcom.it

☐ **Abbonamento Italia** **€ 100,00** (IVA 22% INCLUSA)

☐ Contribuenti e Librerie sconto 10%

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P.I.: * …… ***C.F.:*** * ……

e-mail: *……

(*) **campi obbligatori**

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PER LE INDUSTRIE DEGLI OLI E DEI GRASSI

Innovazione e ricerca

Olive oil proficiency tests

Chemical-physical parameters and contaminants

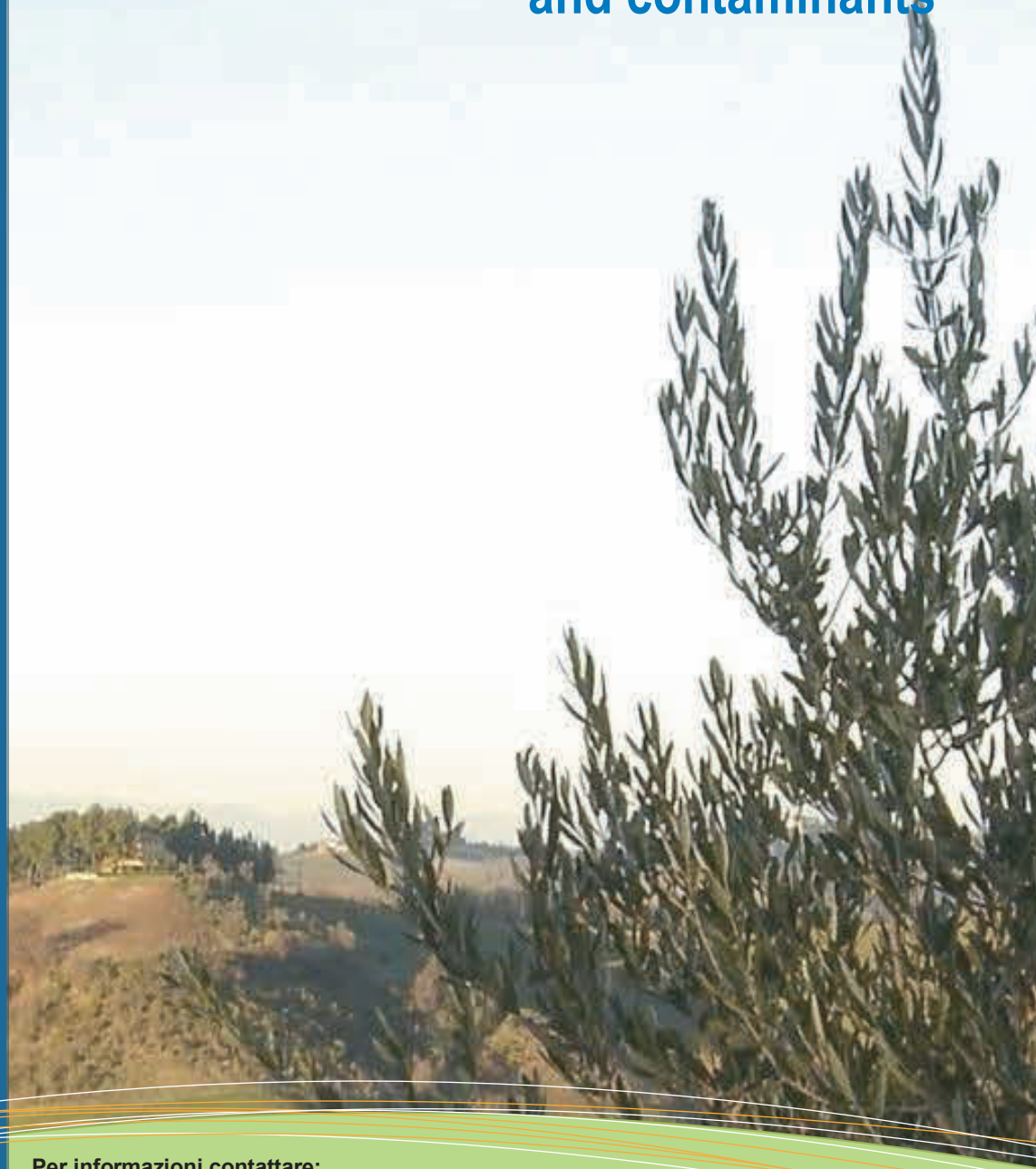
Oil and Fats Laboratory

Since 2003, the Oils and Fats Area, organizes every year an interlaboratory test on olive oil for different commercial categories among various olive oil laboratories.
The tests include all the chemical parameters.
Since 2016 the main contaminants are also considered.

Each participant will have the opportunity to compare his own test results with those obtained by the most accredited Italian and foreign laboratories.

The proficiency test has as main purpose, the ability to make corrections from deviation that might occur in the results, compared to the average value obtained by other laboratories.

At the end of the laboratory tests, the results will be statistically processed and delivered anonymously to each participant.



Per informazioni contattare:

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