

Physicochemical and oxidative stability characteristics of high- and mid-oleic sunflower seed oil

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ABSTRACT

- In South Africa sunflower is the largest source of vegetable oil. Traditionally sunflower oil has been a popular vegetable oil, but the fatty acid composition of standard sunflower oil is far from being ideal for specific uses that require high stability of the oil. Recent research has led to the development of high-oleic acid sunflower varieties with oil that approaches or exceeds 89% oleic acid content. These variants were developed through conventional breeding in the 1980s and 1990s and are speciality oils useful in food products such as spray oils, frying oils and for other products that require oil with high oxidative stability. South African high-oleic sunflower hybrids have been released and seed is commercially available. High-oleic sunflower oil production started in 2003 and is still in the foundation stage and therefore not much information is available on the quality and oxidative stability of South African high- and mid-oleic sunflower oil. The aim of this study was to evaluate and compare the physicochemical properties along with the oxidative stability of traditional and high- and mid-oleic sunflower oils developed and grown in South Africa.
- Total lipid from ground seed samples of traditional, mid-oleic and high-oleic acid sunflower hybrids were quantitatively extracted using a Soxhlet extractor. Fatty acids were determined as methyl esters by gas chromatography. The physical and chemical characteristics of oil samples were evaluated by determination of the refractive index, iodine value and free fatty acid content. Oxidative stability was evaluated by measuring peroxide and *p*-anisidine values. Oil stability index (OSI) measurements were done using the oxidative stability instrument from Omnion.
- The oleic acid contents of the high-oleic hybrids were on average 82.6%, the mid-oleic hybrids 53.8% and the traditional hybrids 22.9%. The refractive index and iodine values were positively correlated with the unsaturation levels of oil samples and were indicative of the oil's susceptibility to oxidation. The oil of the high-oleic sunflower hybrids proved to be of good oxidative quality with low free fatty acid content and low peroxide and *p*-anisidine values. High-oleic sunflower oil had considerably better oxidative stability than traditional sunflower oil. The high-oleic sunflower oil was the most stable of the three oil types with an average OSI of 8.15 h, followed by the mid-oleic oil (4.10 h) and traditional sunflower oil (2.70 h).
- High-oleic sunflower seed oil has superior oil quality and oxidative stability properties compared to traditional sunflower oil and the South African hybrids will meet the demands for more healthy and stable cooking oil.

Key words: fatty acids, *Helianthus annuus* L., oil stability index, peroxide value

INTRODUCTION

Sunflower (*Helianthus annuus* L.) oil is the major product of seed processing that contributes approximately 95% to the total value of the crop. The oil is used mainly as a salad oil and cooking medium, but industrial applications include use as a frying oil and the manufacture of mayonnaise, margarine, shortening and other products (Grompone, 2005). In order to be used for these applications, the oil is subject to specific quality requirements. In the past decades plant breeders have made great efforts to improve sunflower for oil quality features that are mainly related to the fatty acid composition. Traditional sunflower oil with its high polyunsaturated fatty acid (linoleic acid) content and relatively low total saturated fatty acid content has excellent nutritional properties. However, sunflower oil is often judged unsuitable for continuous deep-frying because of its high unsaturation level and susceptibility to oxidation (Salunkhe et al., 1992). Lipid oxidation is an important reaction that can cause deterioration in the quality of edible oil. The consequences of lipid oxidation are the formation of off-flavours and odours, associated with rancidity, as well as loss of functional and nutritional value (Grompone, 2005). Traditionally some oils need to be hydrogenated to enhance their oxidative stability, but partial hydrogenation leads to the production of *trans*-fats that are a health concern (Gupta, 2002; Stender and Dyerberg, 2004). Fortunately plant breeders have been successful in overcoming limitations of the traditional oil by developing a wide range of novel and healthier oil types (Fernández-Martínez et al., 2004). Recent research has led to the development of high-oleic acid sunflower varieties with oil that exceed 89% oleic acid content. The high monounsaturations makes high-oleic sunflower oil much less susceptible to oxidative degradation than traditional sunflower oil with high polyunsaturation (Dorrell and Vick, 1997). As a result high-oleic oil is naturally stable and does not need to be hydrogenated.

In South Africa sunflower is the largest source of vegetable oil. It contributes about 80% to the total oil produced and is followed by soybeans and canola that make up the balance. During the last few years sunflower seed production in South Africa has shown an increase from 300000 ton in 2007 to 860000 ton in 2011 (Dredge, 2011). The development of healthier sunflower oil types was encouraged by South African breeding companies after breeders rights in America expired. Breeding for high-oleic acid sunflower started around 1983 in South Africa. High-oleic hybrids have been released and seed is commercially available. However, high-oleic sunflower oil production started only 2003 and is still in the foundation stage. The aim of this study was to evaluate and compare the oxidative quality and stability of traditional, high-oleic and mid-oleic sunflower hybrids developed and grown in South Africa.

MATERIALS AND METHODS

The seed analysed in this study were obtained from a PANNAR[®] breeding trial planted in Lichtenburg during the 2008/2009 season. Seed material included three traditional (linoleic), three high-oleic and one mid-oleic hybrid entry with three replications for each entry. Due to confidentiality reasons the names of hybrids may not be revealed. Once received, seed were vacuum-sealed and stored at 4°C until needed for oil extraction. Crude seed oil was extracted from ground sunflower achenes using a Soxhlet extractor with diethyl ether as solvent. The extracted oil samples were left to cool and filtered to remove waxes. The filtered oil was transferred into individual dark bottles and stored with nitrogen in the headspace. Fatty acid methyl esters were prepared for gas chromatographic analysis of the extracted fat (Slover and Lanza, 1979). These were quantified using a Varian GX 3400 flame ionization gas chromatograph. Fatty acids were expressed as the relative percentage of each individual fatty acid as a percentage of the total of all the fatty acids present in the sample.

The physical and chemical properties of the extracted oil samples were analysed by determination of the refractive index (RI), the iodine value (IV), the free fatty acid (FFA) content, peroxide value (PV) and *p*-anisidine value (*p*-An V). The RI of all oil samples was determined according to the Association of Official Analytical Chemists' (AOAC) Official Method 921.08 (2000) with an Abbé automatic digital refractometer from ATAGO[®] (Model RX 5000α). The RI-readings were done in triplicate at a temperature of 40°C. The IV was determined according to AOAC Official Method 920.158 (2000). The IV was expressed as the number of gram iodine absorbed by 100 g of lipid. FFA content was determined according to the method described by Pearson (1973) and was calculated as free oleic acid on a percentage basis. The PV and *p*-An V gave an indication of the initial oxidative status of the extracted oil samples. The PV and *p*-An V were determined according to the AOAC Official Method 965.33 (2000) and the method described by Hamilton and Hamilton (1992) respectively. The oxidative stability of the three sunflower oil types was studied using the Rancimat test. The oxidative stability instrument from Omnion was used according to the American Oil Chemists' Society (AOCS) Official Method Cd 12b-92 (1993). The oil sample was held in a thermostated bath and oxidation was carried out at 110°C. A stream

of purified air was passed through the lipid at a rate of 2.5 ml/s. Determination of the induction period was based on the conductometric detection of volatile organic acids swept from the oxidising oil.

RESULTS

The mean seed oil content varied between 35.65-36.56% among the traditional, high-oleic and mid-oleic oil types (Table 1). Although the traditional hybrids contained on average 0.6% more oil than the high oleic hybrids, the three oil types did not differ significantly for oil content. The traditional hybrid 3 contained the highest percentage of oil (38.08%) and this was significantly higher than the oil contents for the traditional hybrid 1 (34.46%) and the high-oleic hybrid 6 (34.66%). The mid-oleic hybrid 7 had a similar oil content to the traditional hybrids, but was not significantly different from any other hybrid.

Table 1. Mean oil content and fatty acid composition in traditional, high-oleic and mid-oleic sunflower oil.

Oil type	Hybrid	Oil content (%)	Fatty acid composition (%)							
			C16:0	C18:0	C18:1	C18:2	MUFA	PUFA	SFA	PUFA/SFA
Traditional	1	34.46b	5.91b	3.81a	26.95b	61.96b	27.12b	62.02b	10.86b	5.71a
	2	36.20ab	6.22b	3.83a	22.66c	65.97a	22.82c	66.04a	11.14b	5.93a
	3	38.08a	6.70a	3.89a	19.12c	69.04a	19.26c	69.10a	11.64a	5.94a
<i>Mean</i>		36.25	6.28	3.84	22.91	65.66	23.07	65.72	11.21	5.86
High-oleic	4	35.51ab	3.27c	4.07a	84.08a	6.61c	84.31a	6.71c	8.98de	0.76b
	5	36.78ab	3.25c	3.62a	81.67a	9.45c	81.93a	9.55c	8.53e	1.12b
	6	34.66b	3.68c	4.14a	81.94a	8.28c	82.18a	8.43c	9.39cd	0.90b
<i>Mean</i>		35.65	3.40	3.94	82.56	8.11	82.81	8.23	8.97	0.93
Mid-oleic	7	36.56ab	4.81d	3.57a	53.77d	36.37d	53.95d	36.42d	9.63c	3.78c

C16:0: Palmitic acid, C18:0: Stearic acid, C18:1: Oleic acid, C18:2: Linoleic acid, MUFA: Monounsaturated fatty acids, PUFA: Polyunsaturated fatty acids, SFA: Saturated fatty acids. Mean values followed by the same letter did not differ significantly at $P \leq 0.05$.

The predominant fatty acids detected were palmitic (C16:0), stearic (C18:0), oleic (C18:1c9) and linoleic acid (C18:2c9,12). Palmitoleic (C16:1), α -linolenic (C18:3c9,12,15), arachidic (C20:0), eicosenoic (C20:1c11), eicosadienoic (C20:2c11,14), behenic (C22:0) and lignoceric (C24:0) acid were detected in minor percentages in all oils, while vaccenic acid (C18:1c7) was only detected in the traditional and mid-oleic oils. Insignificant percentages of myristic acid (C14:0) were detected in a small number of oil samples. Only major fatty acids palmitic, stearic, oleic and linoleic are presented and discussed (Table 1). Averages for palmitic acid content ranged between 3.40-6.28% among the three oil types. Significant differences were observed between oil types for palmitic acid content. The traditional oil contained on 2.88% and 1.47% more palmitic acid than the high-oleic and mid-oleic oils respectively. A highly significant and negative correlation was observed between palmitic and oleic acid (Table 2). Stearic acid content ranged between 3.57-3.94% and did not vary significantly between the three oil types. A significant and negative correlation was observed between stearic acid and oil content (Table 2), but was evident only in the high-oleic hybrids. The mean linoleic acid content of the traditional hybrids (66%) was significantly higher than for the high-oleic (8%) and mid-oleic hybrids (36%). The mean oleic acid content of the high-oleic hybrids (83%) was significantly higher than for the traditional (23%) and mid-oleic (54%) hybrids. The increase in oleic acid content and corresponding decrease in linoleic acid content was due to the significant and negative correlation between oleic and linoleic acid content (Table 2). The traditional hybrid 1 differed from the other two traditional hybrids with significantly higher oleic and lower linoleic acid contents respectively. No significant differences were observed between the high-oleic hybrids for oleic and linoleic acid content.

The three oil types differed significantly in their RI and IV values due to the significant variation in their fatty acid composition (Table 3). Both RI and IV showed significant and positive correlations with linoleic acid and PUFA content (Table 2). As a result, the traditional hybrids had the highest RI and IV values, followed by the mid-oleic and high-oleic hybrids (Table 3). For RI significant differences were observed among some hybrids within each oil type that could be attributed to the significant differences in their linoleic acid contents. The traditional and mid-oleic oil did not differ significantly for IV, but both were significantly different from the high-oleic oil (Table 3). No significant differences were observed

among the hybrids within each oil type for IV. The RI and IV values were in the ranges recommended by the Codex standards. FFA content ranged between 0.32-0.62% (Table 3) for all three oil types.

Table 2. Significant correlations between oil content, fatty acids and physicochemical properties.

	Oil content	C16:0	C18:0	C18:1	C18:2	SFA	MUFA	PUFA	PUFA/SFA	RI
C16:0	0.29									
C18:0	-0.65*	-0.21								
C18:1	-0.20	-0.98**	0.15							
C18:2	0.21	0.98**	-0.16	-1.00**						
SFA	0.07	0.94**	0.15	-0.94**	0.93**					
MUFA	-0.20	-0.94**	0.15	1.00**	-1.00**	-0.94**				
PUFA	0.21	0.98**	-0.16	-1.00**	1.00**	0.93**	-1.00**			
PUFA/SFA	0.21	0.98**	-0.21	-0.99**	0.99**	0.91**	-0.99**	0.99**		
RI	0.23	0.98**	-0.19	-0.99**	0.99**	0.92**	-0.99**	0.99**	0.99**	
IV	0.20	0.91**	-0.25	-0.94**	0.94**	0.82**	-0.94**	0.94**	0.96**	0.94**

**P \leq 0.01, *P \leq 0.05, C16:0: Palmitic acid, C18:0: Stearic acid, C18:1: Oleic acid, C18:2: Linoleic acid, SFA: Saturated fatty acids, MUFA: Monounsaturated fatty acids, PUFA: Polyunsaturated fatty acids, RI: Refractive index, IV: Iodine value.

When considering the oxidation indexes, the three oil types differed significantly for average PV (Table 3). The high-oleic hybrids had significantly lower PVs than the traditional and mid-oleic hybrids. No significant differences were observed between the high-oleic hybrids for PV values, but among the traditional hybrids, hybrid 3 had a significantly higher PV than the other two hybrids. This observation was a consequence of the significantly higher linoleic acid content observed for hybrid 3. The PV of the mid-oleic oil (8.18) was significantly lower than that of the traditional oil (13.33). The traditional oil showed on average the highest *p*-An V of 2.97 and was followed by the mid-oleic oil with a *p*-An V of 2.27. The high-oleic oil had the lowest average *p*-An V of 0.50. However, the three oil types did not differ significantly for *p*-An V. No significant differences were observed between individual hybrids for *p*-An V apart from the traditional hybrid 1 that had a significantly higher *p*-An V than all three high-oleic hybrids. Only hybrids with PVs of 10 meq/1000 g and less (Table 3) were used for oxidative stability analysis and therefore only hybrids 1, 4, 5, 6 and 7 were included for OSI determination. A PV of less than 10 meq/1000 g indicates that oil oxidation has not occurred yet and therefore that the oil is of good oxidative quality. Only the high-oleic hybrids were statistically analysed for variation because there were three entries for this oil type. The traditional and mid-oleic oils contained only one entry each and could therefore not be analysed for variation.

The traditional, mid-oleic and high-oleic oils differed in their OSI times (Table 3). This observation was a result of the different unsaturation levels of the oils. The traditional hybrid 1 showed the lowest OSI value (2.70 OSI/h) and was followed by the mid-oleic hybrid 7 that showed a slightly higher value (4.10 OSI/h). The high-oleic hybrids had the highest OSI values with a mean of 8.15 OSI/h. Among these, hybrid 4 performed the best of all hybrids with the highest OSI value (10.38 OSI/h) that was followed by hybrid 5 (9.02 OSI/h) and hybrid 6 (5.05 OSI/h). Although the three high-oleic hybrids differed in their OSI values, the differences were non-significant.

Table 3. Mean values for physical and chemical tests of traditional, high-oleic and mid-oleic sunflower oil types.

Oil type	Hybrid	Refractive index (N _D 40°C)	Iodine value	Free fatty acid (% oleic acid)	Peroxide value (meq/1000 g)	<i>p</i> -Anisidine value	OSI at 110°C (hours)
Traditional	1	1.467142b	105.64a	0.32a	10.33bc	4.55a	2.70
	2	1.467563a	102.23a	0.37a	12.32b	2.15ab	-
	3	1.467824a	104.81a	0.41ab	17.34a	2.22ab	-
<i>Mean</i>		1.467510	104.23	0.37	13.33	2.97	-
High-oleic	4	1.462098d	83.86b	0.32a	3.17d	0.44b	10.38a
	5	1.462420c	86.78b	0.33a	4.10d	0.78b	9.02a
	6	1.462090d	85.29b	0.62b	3.19d	0.27b	5.05a
<i>Mean</i>		1.462203	85.31	0.34	3.49	0.50	8.15
Mid-oleic	7	1.464854e	101.33a	0.32a	8.18c	2.27ab	4.10

Mean values followed by the same letter did not differ significantly at P \leq 0.05. N_D: Refractive index measurement value at 589.3 nm (the D Fraunhofer line), -: missing values.

DISCUSSION

The high- and mid-oleic sunflower hybrids had oil contents similar to that of traditional sunflower hybrids. The mean seed oil content of approximately 36% for the three oil types was relatively low compared to oil contents reported previously (Rondanini et al., 2003; Radić et al., 2008). Oil content in sunflower seed ranges between 25-48%, but can reach 65% depending on the genotype and environmental factors (Salunkhe et al., 1992; Weiss, 2000). The lower oil contents observed could be attributed to genetic background differences and environmental influence. Unfavourable conditions like heat or drought stress occurring particularly during seed development may have resulted in reduced oil yield (Flagella et al. 2002; Göksoy et al., 2004; Rondanini et al., 2003). The high-oleic, mid-oleic and traditional sunflower hybrids differed significantly in their fatty acid contents. The traditional hybrids (PUFA oil) contained a high linoleic acid (66%) and low oleic acid content (23%) the mid-oleic hybrid 53.77% oleic acid (36.37% linoleic acid) and the high-oleic hybrids (MUFA oil) 82.56% oleic acid (8.11% linoleic acid). The high-oleic hybrids contained 3% less palmitic acid than the traditional hybrids. This negative correlation between palmitic and oleic acid was genetic and contributed to the lower SFA content observed in the high-oleic hybrids. This tendency is generally observed for high-oleic sunflower oil (Martín-Polvillo et al., 2004; Márquez-Ruiz et al., 2008; Merrill et al., 2008).

The PUFA/SFA ratios as well as RI and IV values were indicative of unsaturation levels and as a result oils' tendency to undergo autoxidation (Farhoosh et al., 2008). Decreased levels of unsaturation (linoleic acid) will result in increased levels of oxidative stability. Therefore the high- and mid-oleic sunflower oils with their lower levels of unsaturation should be more resistant to oxidation than the traditional sunflower oil. This conjecture was verified by determining the oxidative quality and stability of the three oil types. The FFA content is an important oil quality parameter (Moschner and Biskupek-Korell, 2006). The FFA values obtained were below the limit of 2% and indicated that the oil of all seven hybrids was of good oxidative quality. The initial oxidative status of the three oil types was evaluated by measuring PV and *p*-An V values. The significantly lower PV and *p*-An V values observed for the high-oleic sunflower oil confirmed that this oil type was more stable to oxidation than the traditional sunflower oil. For the traditional oil, autoxidation already started during seed storage and was in the induction period. High-oleic sunflower oil has longer induction periods than traditional sunflower oil (Martín-Polvillo et al., 2004). The oxidative stability of the three oil types were assessed under accelerated oxidation conditions, using the OSI test. Since OSI measures the resistance of lipids to oxidation, OSI duration is positively associated with oil stability (Guillen and Cabo, 2002). The high-oleic sunflower oil was the most stable oil with the highest OSI value, while the mid-oleic oil also showed better oxidative stability than the traditional sunflower oil. The considerably better oxidative stability of the high- and mid-oleic sunflower oil was attributed to its low level of unsaturation. Márquez-Ruiz et al. (2008) and Merrill et al. (2008) also reported lower OSI values for traditional sunflower oil compared to high-oleic oil. Among the three high-oleic acid hybrids analysed, hybrid 4 showed the best initial oil oxidative status and had the best resistance to oxidation. This hybrid should be considered for release in commercial production as high-oleic acid hybrid with excellent oil oxidative stability.

In conclusion, the extent of variation obtained through traditional breeding for sunflower seed oil quality is evident when comparing the fatty acid composition of the three oil types for fatty acid composition and physicochemical properties. The oil of the high-oleic hybrids proved to be of good oxidative quality and stability with low FFA content, PV and *p*-An V values. Oil of the high-oleic sunflower hybrids showed good oil quality and excellent oxidative stability compared to oil of traditional sunflower hybrids. The South African high-oleic hybrids with oleic acid contents of above 80% meet the demand for more healthy cooking oil that has superior oxidative quality and storage stability properties.

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