SUNFLOWER OIL QUALITY SYMPOSIUM

AGRONOMIC PERFORMANCE OF SUNFLOWER (HELIANTHUS ANNUUS L.) IN AN ORGANIC CROP ROTATION SYSTEM IN THE HUMID TROPICS

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ABSTRACT

The demand for organic sunflower seeds is very high in the international market. Sunflower is a rustic plant that is cultivated under different production systems across several agro-ecological zones in the world. A locally adapted and late maturing sunflower variety ('Funtua') was sown after soybean, sesame and maize between 2008 and 2012 to assess its agronomic performance under continuous, rotational and conventional cropping systems in the forest – savanna transition zone. The field trials were carried out during the late cropping season (June – Nov.) in a randomized complete block design and replicated four times. Data were collected on plant height at maturity, seed yield and yield attributes of sunflower each year. Varying results were obtained on the effects of cropping systems on the agronomic parameters measured across the years. However, cropping system significantly (*P<0.05; F-test*) affected seed yield of sunflower in 2009, 2011 and 2012. The conventional cropping system only significantly (*P<0.05*) produced seed yield (1642.6 $kg.ha^{-1}$) higher than the continuous (778.0 $kg.ha^{-1}$) and rotational cropping (1262.0 $kg.ha^{-1}$)systems in 2009. Thereafter, as the system stabilized, the rotational cropping system recorded higher seed yield than the continuous and conventional cropping systems in 2010, 2011 and 2012. The difference was significant (*P<0.05*) in 2012 with the rotational cropping system producing seed yield higher by 7.3 and 31.3% than the conventional and continuous cropping systems, respectively. Adoption of rotational cropping system is hereby recommended for sustainable organic crop production system in the humid tropics.

Key words: crop rotation, sesame, sunflower, yield, yield characters

INTRODUCTION

Sunflower (*Helianthus annuus* L.) is an oilseed crop that has a very wide range of adaptation ability, low labour requirement for its cultivation and also very suitable for mechanization (Ozer *et al.,* 2004; Kaemeini *et al.,* 2009). Consequently, sunflower can be described as a suitable crop for crop rotation scheme in the tropics where water and not temperature is the major growth limiting factor. It also exhibits erect growth habit, comparable resistance to lodging, short duration, limited ground cover and has easily harvestable heads (Robinson, 1984; Kamal and Bano, 2009). Sunflower is grown principally for its seed that contains oil (36–52%) and protein (28–32%) as reported by Rosa *et al.* (2009). According to NSA (2016), the world's average yield and total land area statistics of sunflower increased appreciably by 11.3% and 9.3% between 2007/2008 and 2012/2013, respectively.

Crop rotation is a planned order (sequence) of specific crops from different genus, species, subspecies or varieties on the same field over a given period of time (Helm, 1993). The advantages of crop rotation include: prevention of soil depletion; improvement of soil fertility,

internal resource utilization; reduction of soil erosion, reliance on synthetic chemicals, allelopathic or phytotoxic effects and environmental impact; control of diseases and pest infestation; enhancement of workload distribution and distribution of economic risks (Helm, 1993). According to Kamal and Bano (2009), over 200 natural allelopathic compounds have been discovered and isolated from different cultivars of sunflower. However, the responses of crops that follow sunflower in a sequence of as companion crops vary (Farooq *et al*., 2011; Nikneshan *et al.*, 2011). It was recently reported that the α-pinene in essential oil of sunflower head is very critical to the inhibitory effect of head extract (Kaya *et al.*, 2013). Consequently, it was suggested that removing the head of sunflower could be beneficial for alleviating the allelopathic effect. Unfortunately, this crop is rarely cultivated in rotation with other crops in the tropics. Therefore, in a bid to develop a production package for some staple and commercial food crops with high export potentials a crop rotation scheme was initiated consisting of four component crops with export potentials (soybean, sunflower, sesame and maize) in 2008. The objective of the study was to evaluate the performance of the component crops in rotation relative to continuous and conventional cropping systems.

MATERIALS AND METHODS

The mean monthly rainfall data during the late copping season of 2008 – 2009 are presented in Table 1. Year 2010 was the wettest year (791.2 mm) during the late cropping season and 2008 was the driest (328 mm). Although, the highest rainfall (288.1 mm) was recorded in 2011 during the most critical month for sunflower (October) which coincided with grain filling. The crop rotation scheme involved four component crops (soybean, sesame, sunflower and maize as shown in Table 2) and the study was carried out at the Organic plot of the Teaching and Research Farm of the University of Agriculture, Abeokuta (7º 15' N, 3º 25' E, altitude 140 m.a.s.l). The soil of the experimental field is oxic Paleudulf (Adetunji, 1991). The test variety of sunflower was Funtua (a local adapted and late maturing variety). The experimental design was randomized complete block design (RCBD) with four replicates. Treatments evaluated were continuous, rotational and conventional cropping systems. The plots of the conventional cropping system were located about 15 m away from the organic plots to avoid commingling. The row spacing adopted for sunflower under the three cropping systems was 60 x 30 cm and each plot measured 6.5m by 6.0m ($39m^2$). Sowing of sunflower seeds was done on August 15, 2008, July 2, 2009, August 15, 2010, July 18, 2011 and July 20, 2012 based on the onset of rains in the late cropping season. No herbicides or inorganic fertilizers were applied on the continuous and rotation plots. However, pre-emergence herbicides (Galex and Gramoxone) and fertilizer combination (60 kgN/ha, 56 kg P_2O_5/ha and 100 kgK_2O/ha) were applied on the conventional plots at sowing and 4 weeks after sowing (WAS), respectively. Manual weeding was done on all plots at 3 and 6 WAS. The organic fertilizer (Aleshinloye Fertilizer (Grade B) contained 1.2%N, 76 ppm P, 13.75 cmol K, 10.28 cmol Na) was applied at the rate of 25 tonnes/ha to the continuous and rotational cropping systems plots at 4 WAS. This rate was equivalent to 60 kg N ha/ha of the inorganic fertilizer recommended for sunflower in the transition zone (Olowe *et al.,* 2005). Application of organic fertilizer commenced in 2009 a year after the rotation scheme took off. Harvesting was done at physiological maturity (R8) as described by Schneiter and Milner (1981). Five randomly selected plants per plot were tagged from the net plot for plant height measurement and yield attribute analysis. Data were collected on plant height at physiological maturity, head weight and diameter, number and weight of seeds per head and seed yield on plot basis. All data collected were subjected to analysis of variance and means of significant treatment were separated using the least significant difference method as described by Steel and Torrie (1984).

RESULTS

Effect of cropping systems on plant height, seed yield and yield attributes of sunflower

Cropping system only significantly ($P \leq 0.05$; *F*-test) affected plant height in 2012 with sunflower plants on rotational and conventional plots significantly taller than plants on under continuous cropping system (Table 3). However, the pooled mean indicated that the plant height of sunflower under the conventional and rotational cropping systems were at par. Average head diameter and weight of sunflower were significantly ($P \leq 0.05$; *F*-test) affected by cropping system in 2009 and when pooled, and the plants under continuous cropping system recorded significantly lower head diameter and weight than those under rotational and conventional cropping systems (Table 4 and 5). Weight of seeds per head was significantly ($P \leq 0.05$; *F*-test) affected by cropping system in 2009, 2012 and when pooled (Table 6). Similarly, the effect of cropping system was only significant ($P \leq 0.05$; *F*-test) for number of seeds per head in 2009 and when pooled (Table 7).However, sunflower seed yield was significantly ($P \leq 0.05$; *F*-test) affected by cropping system in 2009, 2011, 2012 and when pooled. Sunflower under continuous cropping system produced lower (significant at $P \leq 0.05$) seed yield than the plants under rotational and conventional cropping systems during the three years, except when yield values were pooled and the continuous was at par with rotational system (Table 8).

DISCUSSION

Rainfall distribution which is the main growth limiting factor in tropical agriculture varied markedly during the five year period of experimentation. The total rainfall during the late cropping season of 2008 – 2012 ranged between 328.0 and 791.2 mm and these values compared favorably with the rainfall amount (500 – 750 mm) reported to be adequate for optimum performance of sunflower (Weiss, 2000). Year 2008 with the smallest amount of rainfall (328.0 mm) also recorded the lowest seed yield (540.8 kg/ha). This could be attributed to the low rainfall in October (84.5 mm) which coincided with the grain filling period. The rotational and conventional cropping systems recorded grain yields above 1,000 kg/ha between 2009 and 2012, except conventional cropping system in 2010 and 2011. These yield values must have been enhanced by the rainfall in the months of September and October, and the availability of nutrients supplied through fertilizer application and they compared favorably with Nigerian (1000 kg/ha), African (812 kg/ha) averages (Olowe *et al.*, 2013) and world average (1520 kg/ha) according to USDA (2012), and the more recent forecast (1410 kg/ha) for 2012/2013 by NSA (2016). The consistently higher seed yield recorded under rotational cropping system in 2010, 2011 and 2012 could be due to the gradual stabilization of the system following application of organic fertilizers and rotation of soybean and sesame as preceding crops to sunflower.

The main agronomic traits that critically contribute to seed yield of sunflower include number of heads per hectare, weight of seeds per head and number of seeds per head (Robinson, 1978). However, in our study, the pooled mean revealed that cropping system significantly affected grain yield with the conventional and rotational cropping systems recording higher values for weight and number of seeds per head relative to sunflower under continuous copping system. Furthermore, the relatively lower values for plant height, number and weight of seeds per head, head weight and diameter on sunflower under continuous cropping system could also be attributed to depleted nutrients in the soil and accumulation of pest and disease organisms following continuous cropping of sunflower for the fourth year on the same plot. However, no serious disease or pest problem was recorded during our study.

CONCLUSION

Based on the pooled results of this study, the agronomic performance of sunflower that received organic fertilizer under rotational cropping system confirmed the huge potential for sunflower being a crop with high adaptability and low labour requirement as a viable component in organic crop rotation system in the tropics.

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Table 1: Mean monthly rainfall (mm) during the late cropping season (July – November) of 2008 - 2012

Year	July	August	September	October	November	Total
2008	299.2	106.7	136.8	84.5	0.0	328.0
2009	160.0	162.1	151.6	180.1	64.6	718.3
2010	322.9	266.6	257.6	172.3	94.7	791.2
2011	349.5	88.7	204.1	288.1	3.6	584.5
2012	155.4	36.3	181.4	184.7	49.6	607.4

Table 2: Crop rotation scheme involving soybean, sesame, sunflower and maize (2008 -2012)

2008	2009	2010	2011	2012
Sunflower	Sesame	Maize	Soybean	Sunflower
Sesame	Soybean	Sunflower	Maize	Sesame
Maize	Sunflower	Soybean	Sesame	Maize
Soybean	Maize	Sesame	Sunflower	Soybean

Table 3: Effect of cropping systems on plant height (cm) of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	2008	2009	2010	2011	2012	Mean
Continuous	-	184.7	125.8	206.1	218.0	183.7
Rotational	208.0	209.0	224.3	255.8	235.0	226.4
Conventional	192.0	206.4	216.7	243.7	237.8	219.3
LSD 5%	ns	ns	ns	ns	10.19	32.32

Notes: **, * Significant at P ≤ 0.001 and 0.05, respectively, ns – non-significant

Table 4: Effect of cropping systems on head diameter (cm) of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	2008	2009	2010	2011	2012	Mean
Continuous	-	9.6	9.1	14.1	17.1	12.5
Rotational	9.8	12.1	10.5	16.2	18.0	13.3
Conventional	8.6	12.8	11.3	16.4	18.1	13.4
LSD 5%	ns	2.17	ns	ns	ns	0.69

Notes: **, * Significant at P ≤ 0.001 and 0.05, respectively, ns – non-significant

Table 5: Effect of cropping systems on head weight (g) of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	2008	2009	2010	2011	2012	Mean
Continuous	-	32.4	39.9	28.5	112.5	53.3
Rotational	60.3	58.0	57.2	41.3	123.5	68.1
Conventional	43.4	68.0	79.1	41.6	122.5	70.9
LSD 5%	ns	26.50	ns	ns	ns	13.36

Notes: **, * Significant at P ≤ 0.001 and 0.05, respectively, ns – non-significant

Table 6: Effect of cropping systems on seed weight (g) of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	2008	2009	2010	2011	2012	Mean
Continuous	-	20.2	21.9	19.7	41.3	25.8
Rotational	21.5	33.1	31.9	37.9	57.7	36.4
Conventional	28.3	42.2	35.1	36.2	53.4	39.1
LSD 5%	ns	9.53	ns	ns	3.02	9.93

Notes: **, * Significant at P ≤ 0.001 and 0.05, respectively, ns – non-significant

Table 7: Effect of cropping systems on number of seeds per head of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	2008	2009	2010	2011	2012	Mean
Continuous	-	319.0	580.0	385.0	580.5	466.0
Rotational	540.8	680.0	853.0	547.0	607.0	645.4
Conventional	664.9	715.0	659.7	520.0	591.5	630.2
LSD 5%	ns	257.2	ns	ns	ns	57.44

Notes: **, * Significant at P ≤ 0.001 and 0.05, respectively, ns – non-significant

Table 8: Effect of cropping systems on seed yield (kg/ha) of sunflower during the late cropping season (July – Nov.) in 2008 - 2012

Cropping systems	**2008**	**2009**	**2010**	**2011**	**2012**	**Mean**
Continuous	-	778.0	1000.0	584.7	981.1	835.9
Rotational	540.8	1262.0	1150.0	1348.5	1428.2	906.0
Conventional	664.9	1642.6	750.0	808.9	1324.0	1038.0
LSD 5%	ns	366.75	ns	579.8	75.23	145.0

Notes: **, * Significant at P $\leq$ 0.001 and 0.05, respectively, ns – non-significant

LESSONS FROM TEN YEARS OF AN INTERPROFESSIONAL SURVEY PLAN ON OILSEEDS FOOD SAFETY

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ABSTRACT

French oilseeds food chain operators are coordinated through a food safety survey plan, in order to get a realistic picture of the contamination in oilseed products (seeds, oilseed meal, and vegetable oil). Concerned crops are those cultivated or processed in France: rapeseed, sunflower and soybean. Grain storage companies, feeding industries and oil industries participate voluntarily, and send their self-data that are pooled in a database. Thirty-three companies are actively involved, providing each year about 60000 to 180000 analytical results coming from about 2000 to 3000 samples of seeds, meals and oils (note: on one sample, several contaminants can be analyzed giving several analytical results). Pesticide residues represent more than 90% of the analytical results of this database as the laboratories can determine a large number of active substances with multi-methods. Other sought contaminants are: trace elements (cadmium, lead, arsenic, mercury, fluorine), mycotoxins (mainly aflatoxin B1 and total aflatoxins), toxic organic compounds (polycyclic aromatic hydrocarbons, dioxins and PCBs), microbiological contaminants (salmonella in meals), botanical impurities (eg seeds of *Datura spp.* in sunflower seeds), mineral oils or compounds likely to be formed during refining such as esters of 3-MCPD and glycidyl esters in oils. The food safety of oilseeds survey plan allows to identify which are main concerns, for instance post-harvest insecticide residues from cross contamination during storage. Results of this monitoring plan were transmitted to the French government and the European Commission in cases of regulatory threshold revisions (eg for cadmium in oilseeds, for the revision of pirimiphos-methyl thresholds).

Key words: Oilseeds, vegetable oil, survey plan, contaminants, pesticide residues

INTRODUCTION

The French oilseed food supply chain got together with food safety issues since the early 2000s that correspond to the establishment of a set of European regulations called "Hygiene Package" (Dauguet et al, 2006). In this context, the food safety survey plan (called PSO) was implemented from the 2005 campaign, helping to control the quality of products (seeds, meal and oil) in a interprofessional framework. Since PSO was launched, more and more operators of the oilseed supply chain have become active partners. This article gives a review of the seven years of the PSO.

Today, each operator of the food chain is facing a legal obligation:

- to implement a HACCP approach, based on sound analysis of health risks inherent in its business,
- to ensure the sanitary compliance of products that it puts on the market,

- to carry out self-monitoring.

The PSO, set up by Terres Inovia, ITERG and Terres Univia since 2005, is an observatory of the sanitary quality of oilseed products in France (Lacoste et al, 2005). This survey plan is based on a shared private database on oilseed contaminants. This base is fed by self-monitoring data from industries (crushing industry and feed industry) and storage agencies that join this PSO, as well as by series of analyzes on seeds, meal and oil by Terres Inovia, ITERG and Terres Univia (figure 1).

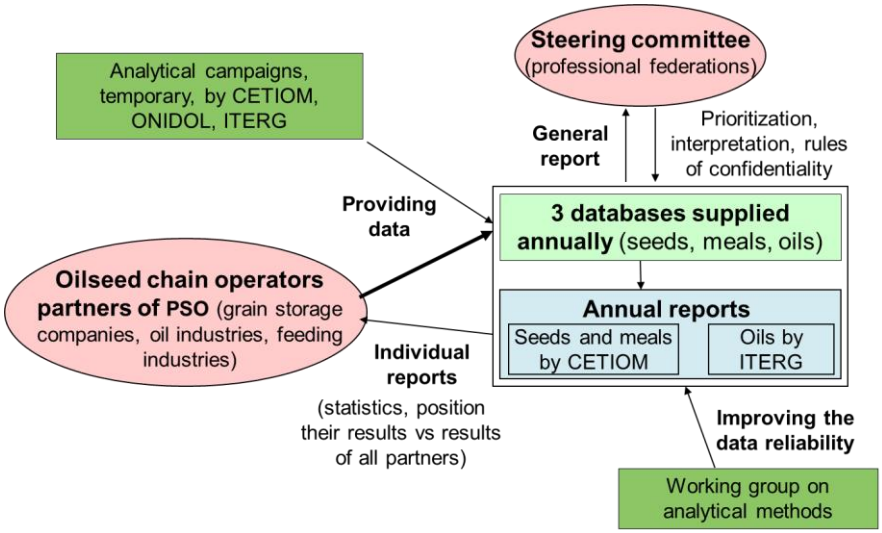

Figure 1. Organization of the Oilseed Survey Plan (PSO)

Intended for storage agencies to industrial oil processors and feed manufacturers, the PSO deals with:

- oilseeds: rapeseed, sunflower, soybean
- products: seeds, meals, crude and refined oils, byproducts of refining
- contaminants: residues of plant protection products, trace metals, mycotoxins, toxic organics, salmonella, botanical impurities ...

The confidentiality of data is guaranteed for partners, and no commercial exploitation of this database is made. The database on seeds and meals is managed by Terres Inovia, and the database on crude and refined oils is managed by ITERG.

So, the PSO is a tool of the oilseed supply chain, allowing a collective coordination on the safety aspects, highlighting progress and contributing to setting realistic regulatory thresholds. It represents also a forum for exchange of information between the operators in the sector, where are identified relevant research avenues.

A GOOD REPRESENTATIVENESS

To date, the PSO has 33 active partners: 28 grain storage agencies distributed throughout France, which represents 30-40% of the of the French oilseed harvest, 4 oil industrials (the main groups in France) and 1 partner in the feed industry, the OQUALIM association, which brings together 57 feeding companies (over 71% of the feed production). The representativeness of the PSO partners is correct. This plan is open to all interested companies and new members join it every year. Thus, each partner provides analysis data from its own self-monitoring data, and annually receives an individual report with its results compared to regulatory limits and to the overall PSO results: a moderate analytical investment gives access to a rich database, allowing refining its risk analysis.

For the last ten years, the PSO collected data annually from about 2,000 to 3,500 samples of seeds, cake, oils, and providing between 40000 and 120000 analytical results per

year (several contaminants checked in each sample). Plant protection products residues (pesticides) represent over 90% of the results. The other investigated contaminants are: metal and mineral trace elements (cadmium, lead, arsenic, mercury, fluorine), mycotoxins (aflatoxin B1 and total aflatoxins essentially), toxic organic (PAHs, dioxins and PCBs), microbiological contamination (salmonella cakes), botanical impurities (seeds of *Datura spp*. in sunflower seeds), mineral oils or compounds likely to form during refining such as the esters of 3-MCPD and glycidol esters in the oil.

THE PSO RESULTS

PSO allows us to check that almost all oilseed products comply with the regulations. Regulatory limits on oilseeds are defined in different texts: maximum limits for pesticide residues (MRLs, EC Regulation No. 396/2005 and Regulations amending it), maximum levels in feed (Directive 2002/32 / EC and texts the modifying) maximum levels in foodstuffs for human consumption (Regulation No. 1881/2006 and other regulations amending it).

However, PSO provides the observation that oil refining is necessary to remove some pesticide residues from crude oils. These are mainly insecticide residues, coming from post-harvest treatments (pirimiphos-methyl, chlorpyrifos-methyl, deltamethrin), applied on the empty storage cells or on cereal grains stored in the same sites, and being incidentally found on oilseeds by cross-contamination (Dauguet, 2007; Dauguet, 2009). These molecules are then removed at various steps of oil refining, and therefore marketed refined oils are pesticide free.

Through the PSO, the real effort provided by crushing plant in order to control the microbiological quality of the meal could be checked: today salmonella nearly disappeared in rapeseed and sunflower meals produced in France.

The PSO suggests that mycotoxins are a danger almost inexistent for oilseeds, considering the regulated toxins. Only aflatoxin can be detected occasionally in sunflower, but at very low levels, far below the regulatory threshold. But monitoring of aflatoxins should as a regulatory threshold for aflatoxins in human food has been fixed for oilseeds intended for direct human consumption (2 mg/kg for aflatoxin B1 and 4 mg/kg for total aflatoxins) without industrial processing (confectionery sunflower), with a much higher maximum levels in feed (20 mg/kg for aflatoxin B1). For other not yet regulated toxins such as toxins of Alternaria, EFSA recommends Member States to acquire data in food. Within PSO, analyzes of these toxins have been carried out and their presence can be seen occasionally on sunflower. However, toxicological studies are not sufficiently substantiated to date to conclude on the risk posed by the toxins of Alternaria.

The trace metals are not a family at risk as oilseeds never exceed these regulatory limits. In the case of cadmium, the concentrations found in the sunflower seeds and meal can be sometimes close to the threshold in animal feed (1 mg/kg).

A contaminant was identified recently in the PSO: *Datura spp* seeds, which are botanical impurities that can be found in sunflower seed crops. This weed is toxic, since it contains tropane alkaloids, and the presence of *Datura spp*. seeds is regulated in the raw materials for animal feed (1000 mg/kg of whole seeds of *Datura*). Indeed, the alkaloids contained in these impurities will be transferred in the meal after the oil extraction process.

Organic toxic substances, such as polycyclic aromatic hydrocarbons (PAHs) and dioxins and PCBs, are specifically monitored in crude and refined oils. The levels measured for these substances show that these substances do not pose a problem in the French oilseed sector.

Recently, the presence of esters of 3-MCPD and glycidol esters has been reported in refined vegetable oils, and in formulated food products containing vegetable fats (Zelinková, 2006). Palm oil is the oil with the highest infection rates likely related to the high temperatures used

during deodorization of physical refining, while the seed oils are generally less prone to the formation of this contaminant (Kuhlmann, 2011). The few results collected via the PSO confirm the low contamination of refined rapeseed and sunflower oils.

Following a sunflower crude oil contamination from Ukraine by mineral oils (Lacoste, 2010), manufacturers have established since 2008 a systematic verification of import sunflower oil. The data collected within the PSO showed that the contamination in 2008 was an isolated case.

PSO, A TOOL FOR THE OILSEED FOOD CHAIN

The results of PSO therefore enable operators in the sector to carry out an analysis of health hazards in oilseed products. Thus, the subject of post-harvest insecticide residues appeared important. This encouraged the operators to carry out specific actions to identify the sources of cross-contaminations of oilseeds by these pesticide residues in storage facilities. Surveys conducted in collaboration with companies have enabled the identification of these situations leading to cross-contamination (Dauguet, 2007; Dauguet, 2009), and recommendations were relayed by the federations. According to the latest PSO results, the contents of these pesticide residues tend to decline.

The PSO has also been involved to argue for re-examine the maximum residue level of pirimiphos-in oilseeds, taking into account the phenomenon of cross-contamination during storage. This data were studied by EFSA which issued an opinion (EFSA, 2011) in which European food safety authority says that an MRL of 0.5 mg/kg would be suitable for oilseeds (while the current MRL was 0.05 mg/kg).

As part of the review of the regulatory thresholds of cadmium in food, PSO partners have also mobilized to provide the public authorities with data so that future limits are not an obstacle to trade in oilseeds. This issue mainly concerns the sunflower, which accumulates cadmium in its seeds. Today, none sunflower sample exceeded the regulatory threshold for feed, but a lower threshold could be a problem. Finally, this regulation does not apply to oilseeds. PSO data were transmitted to the French authorities in the context of the European discussions on the revision of cadmium thresholds, with the agreement of all PSO's members. This also illustrates the value of reliable data to assist in setting realistic regulatory thresholds.

CONCLUSION

The PSO is now considered a sustainable action for the benefit of operators in the French oilseed sector, which has no equivalent in other countries. In 2016, new means of communication and information are available for PSO members, with a dedicated and protected website. This provides more responsiveness and flexibility: more ease for online data entry and data reading.

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THE EFFECTS OF VACUUM AND ATMOSPHERIC DEEP-FAT FRYING PROCESS ON TOTAL FRYING-USE TIME OF SUNFLOWER OIL AND ON FRENCH FRIES QUALITY

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ABSTRACT

Deep-fat frying, which is one of the oldest and popular food preparation methods, is a process of immersing food in hot oil at a high temperature. In this study a vacuum cooking equipment prototype which could work both atmospheric pressure and under vacuum was developed for deep-fat frying process. The effect of vacuum and atmospheric frying temperature and number of frying in the same sunflower oil on the quality of French fries and sunflower oil was evaluated. Potato pieces was fried in ratio 1:6 (potato:oil) at atmospheric pressure and under vacuum at 135 and 180°C, respectively, for 10 min in every frying interval for a total of 7 (atmospheric pressure) and 15 (under vacuum) times of frying in the same oil. The free fatty acid content of the frying oil at atmospheric condition was determined to be excessively high compared to that of vacuum frying oil. TPM of oil at the atmospheric frying after the 3th frying rapidly reached to TPM content of the 15th vacuum frying oil. It was observed that peroxide value of the oil at atmospheric frying was higher than that of vacuum frying oil. Viscosity of the oil at atmospheric condition increased rapidly with an increase in exposure time compared to that of vacuum frying oil. The color values of vacuum and atmospheric fried French fries were not significantly different from each other. No significant changes in texture of French fries were determined with oil utilization time in the both of frying process.

Key Words : Deep-fat frying, vacuum frying, oil utilization time, sunflower oil, oxidation

EFFECT OF CURCUMIN NANOPARTICLES ON OXIDATIVE STABILITY OF SUNFLOWER OIL-IN-WATER EMULSIONS

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ABSTRACT

Curcumin is a natural polyphenolic compound that is obtained from the root of *Curcuma longa* Linn (turmeric). Oil oxidation is an undesirable series of chemical reactions involving oxygen that degrades the quality of oil. The aim of the present study was to develop a method to nano-particularize curcumin in order to increase its antioxidant efficiency against oxidation of sunflower oil. For this purpose, curcumin was dissolved in dichloromethane, injected in heating water (60 ºC) including tween 80 and then stirred. After characterization of the particle size and distribution of the fabricated curcumin nanoparticles, they were lyophilized. In formation of the oil phase of emulsion with nanocurcumin (ENC), nanocurcumin was added into oil-in-water system in which sunflower oil was used as the oil phase. Oxidation stability of oil-in-water emulsions including curcumin nanoparticles was measured by oxidation test reactor. As a result, 98 % of the particles were in mean diameter of 9-10 nm. The formed nanoparticles were characterized by scanning electron microscope, Fourier Transform Infrared Spectroscopy and thermogravimetric analysis. Unlike curcumin, nanocurcumin was found to be freely dispersible in the presence of the surfactant. The chemical structure of nanocurcumin was the same as that of curcumin, and no remarkable change was observed during nanoparticle preparation. Thermal degradation of the nanocurcumin was similar to that of curcumin. It was found that emulsion with nanocurcumin (ENC) was more effective than those with and without curcumin against oxidation of the sunflower oil, as revealed by the longer induction periods (IP) for ENC (1 hr 20 min) than those for emulsions with and without curcumin (60 min. and 53 min.) The results demonstrated that the water solubility and antioxidant activity of curcumin was markedly improved by particle size within the nano-range.

Key Words : Sunflower oil, nanocurcumin, nanotechnology, oxidative stability, molecular and thermal characterization.

DETERMINATION OF TEXTURAL, RHEOLOGICAL PROPERTIES AND SFC, SMP VALUES OF OLEOGELS PREPARED USING SUNFLOWER OIL

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ABSTRACT

In Recent years, food products which is designed to provide development for human health and researches is to improve such products have been intensively carried out all over the World. Oils Reduced Trans and saturated fatty acids levels have come firstly. To this end, oleogels, which have a spreadable elastic structure, by adding organic or polymer gelling agents (oleogelators) to oils, have been used. In our country, sunflower seed provides about 45% our total oil seed production and sunflower oil comes first in mostly consumed edible oils. Oil obtained from sunflower seed is rich in linoleic acid. Also recently, production of high oleic sunflower oil, by reducing linoleic acid content of sunflower oil, has been started. In this study, creating of oleogels formulations include sunflower and high oleic sunflower oil, have low amount of trans and saturated fatty acids, alternate to margarines and determination of textural, rheological, SFC and SMP values of this samples was purposed. For 6 samples (1 reference and 5 new formulations) Textural properties according to Ogutcu and Yilmaz, 2015; rheological properties according to Lupi et. Al., 2013 (with some modifications); SFC values according to AOCS Official Method Cd 16b-93:2009 and SMP values according to ISO 6321:2002 have been proceeding.

Key Words : oleogels, sunflower oil, rhelogical, SFC

AFLATOXIN CONTAMINATION IN SUNFLOWER OIL

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ABSTRACT

Sunflower (*Helianthus annuus* L.) is an annual ornamental herb grown as an oil seed crop. Because of their chemical composition and nutritional value sunflower seeds are considered to be a great source of lipids and proteins and are largely used in the production of edible oils and animal feed. The protein content of the seeds is approximately 50%–60%. Sunflower oil is the preferred oil in most of Europe, East Europe, Russia, Mexico, countries along with Mediterranean and several South American countries. Mycotoxins are poisonous organic compounds produced by several species of fungi. In studies have shown that isolates of different mold species were able to produce aflatoxins B_1,B_2,G_1 and G_2, sterigmatocystin, ochratoxin A, patulin, citrinin, penicillic acid, zearalenone and griseofulvin in sunflower. Aflatoxins are a major group of mycotoxins, which have toxic, carcinogenic and mutagenic activity, causes important health problems and economic losses. The production of oils from oilseeds requires the following steps: storage of grains, preparation, extraction of crude oil and refine (degumming, deacidification, bleaching, deodorization). Some of these steps may be harsh and lead to inactivation or elimination of important compounds, such as vitamins, antioxidants and enzymes, although the effect on undesirable compounds like aflatoxins varies markedly among methods. The high contamination of oilseeds by aflatoxins generates a concern on a global scale due to the high consumption of these products. Several reports have shown high incidences of aflatoxin contamination in plant-derived oils in regions of China, Sudan, India and Sri Lanka. Experimental studies have shown that aflatoxins present in the oleaginous material can be transferred to the final oil product. However, depending on the type processing (extraction and purification) of the crude oil, the levels of these contaminants can be reduced.

Key words: Sunflower oil, mycotoxins, aflatoxins, oil processing

INTRODUCTION

Mycotoxins are secondary metabolites mainly produced by different fungal species when they contaminate food and feed. Various fungal species like such as *Aspergillus*, *Penicillium* and *Fusarium* under different climatic conditions of temperature and humidity can contaminate cereals. Mycotoxin contamination occurs frequently in various food commodities globally, leading to animal and human health risks. More than 400 mycotoxins have been identified and reported and the key important mycotoxins that are highly prevalent in the contaminated agro-food products are aflatoxins, ochratoxin A (OTA), deoxynivalenol (DON), fumonisins (FBs), zearalenone (ZEN), citrinin (CIT) and patulin. Among the types of aflatoxins are aflatoxin B_1, B_2, G_1 and G_2, which are a group of closely related mycotoxins (Selveraj et al., 2015).

Aflatoxin B_1, produced by *Aspergillus flavus* and *Aspergillus parasiticus*, is one of the most toxic and common contaminants in food and feed. Ingestion of aflatoxin-contaminated food leads to acute and chronic toxic effects, which may be hepatocarcinogenic, mutagenic, teratogenic or genotoxic (Samuel et al., 2014).

Aflatoxins are commonly found in nuts, peanuts, corn, cottonseed and other oil seeds which affect not only the health of humans and animals but also the economics of agriculture and food. Aflatoxins are produced under optimum temperature and moisture conditions and cannot be completely avoided, so aflatoxins have become a threat worldwide. In order to protect human safety, limits on aflatoxins have been set in many countries. In the European Commission, the current maximum levels are 2 μg/kg for AFB_1 and 4 μg/kg for total aflatoxins for groundnuts, nuts, dried fruits and cereal (Fan et al., 2013).

Sunflower (*Helianthus annuus* L.) is an annual ornamental herb grown as an oil seed crop (Nahar et al., 2005). Because of their chemical composition and nutritional value sunflower seeds are considered to be a great source of lipids and proteins and are largely used in the production of edible oils and animal feed. The protein content of the seeds is approximately 50%–60%. (Beheshti and Asadi, 2013). Sunflower oil is the preferred oil in most of Europe, East Europe, Russia, Mexico, countries along with Mediterranean and several South American countries.

Sunflower seed contain 25-32 % edible oil which is a rich source of polyunsaturated fatty acids used for human consumption. Sunflower seeds also provide a nutritious food for cattle, poultry hogs and cage birds. The seeds are also consumed roasted, salted and a coffee substitute is prepared from roasted seeds. Of the different fungi isolated from sunflower seed, *Aspergillus flavus* Link. was found to be most predominant (Dawar and Ghaffar, 1991).

The high contamination of oilseeds by aflatoxins generates a concern on a global scale due to the high consumption of these products. Several reports have shown high incidences of aflatoxin contamination in plant-derived oils in regions of China, Sudan, India and Sri Lanka. Experimental studies have shown that aflatoxins present in the oleaginous material can be transferred to the final oil product. However, depending on the type processing (extraction and purification) of the crude oil, the levels of these contaminants can be reduced (Bordin et al., 2014).

SUNFLOWER OIL PROCESSING

Oil processing involves three major conventional processes which include continuous neutralizing, bleaching and deodorisation. Neutralisation of crude oil with caustic soda is still an essential feature for a refinery required to produce a consistently high quality product and to handle a number of different oil types. A bleaching step is necessary to remove soap, trace metals, sulphurous compounds and part of the more stable pigments and pigment breakdown products which have resulted from raw materials damage or oxidation. The deodorization process involves steam distillation under vacuum. Its purpose is to remove so far as possible residual free fatty acids, aldehydes and ketones which are responsible for unacceptable oil odours and flavors and, more recently to decolourise the oil by heat decomposition (270ºC) of the pigments and distillation of the decomposition products (Banu and Muthumary, 2010).

The sunflower oil process flow diagram is shown in Fig. 1 (Pal et al., 2015).

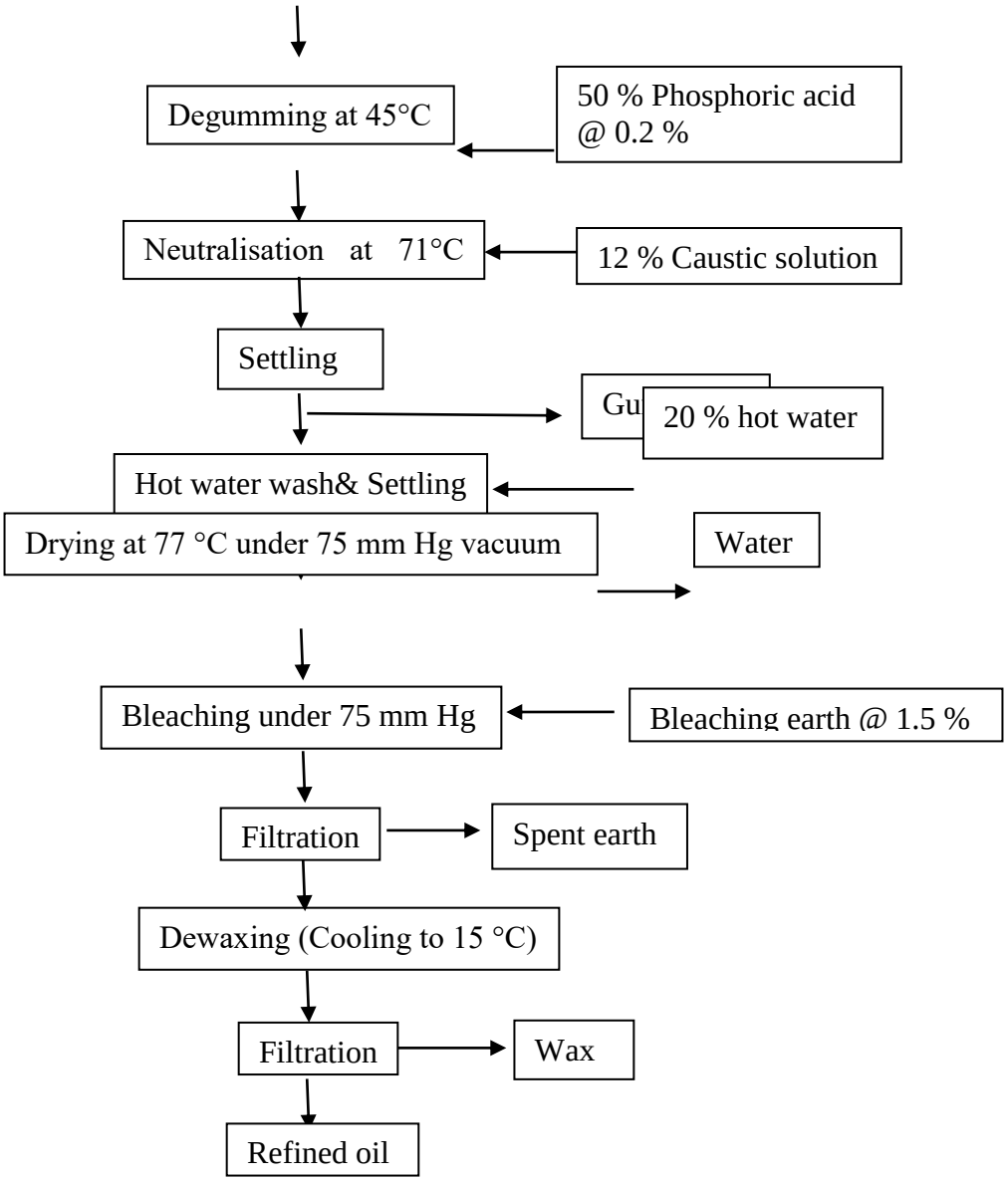

Fig. 1 Process flow diagramme for refining of sunflower oil

SUNFLOWER OIL AND AFLATOXIN

The seeds of sunflower (*Helianthus annuus),* may first have been cultivated by Indian tribes in North America, about 3000 B. C. The seeds were used as a food, medicine and as a religious symbol. The plant was brought by Spanish travelers to Europe sometime around 1500 AD. The sunflower thrives in temperate climate and is today cultivated in USA, Europe, Russia and Canada among others. For the food industry the seeds are largely cultivated for oil production, but the seeds are also used for food or bird feed (National Sunflower Association 2013). Sunflower seeds can contain up to 45% oil (Eklöf, 2013). Sunflower seeds are a good substrate for aflatoxin production. Lipids may play, an important role in the biosynthesis of aflatoxin (Chulze et al., 1990).

The mycoflora of sunflower seeds appeared to be diverse, with many toxin producing species as common contributors. The analysis of toxins did however not detect any toxins, and since

the commodity was storage stable (the highest aw detected was 0.63), there did not seem to be any risk involved with consuming these products. However, if the commodity would be exposed to moisture there would be a risk of toxin production. Abdullah et al. (2010) found common genera in sunflower seeds to be *Aspergillus, Alternaria, Fusarium,* and *Penicillum. A. niger* and *A. flavus* were the most common species, but also *Penicillium expansum* were common. Jimenez et al. (1991) found highest counts of *A. niger* and *Penicillium spp*. and Shahnaz et al. (1991) found high incidence of *A.flavus* and *A. niger.* Sharfun-Nahar et al. (2005) also found along with high incidence of *Penicillium* spp. The study also revealed *A. ochraceus* as a contributor to the mycoflora of sunflower seeds, and both studies found species of *Alternaria* and *Fusarium* (Eklöf, 2013).

The oil extraction step, both by pressing and solvent, is not capable to eliminate the aflatoxins. In fact, the toxin is shared between the phases, remaining in both the oil and defatted meal. The oil extraction with non-traditional solvents has been shown viable, and studies performed with ethanol, and isopropanol have also shown the ability to partially remove aflatoxins from oil seeds. However, a point to be elucidated is the appropriate treatment given to the solvent leading to the elimination of toxins, so that it allows its reuse in the process (Bordin et al., 2014).

There are no studies in the literature that relate the degumming with the presence of aflatoxins. However, it is known that the toxins are soluble in polar organic solvents. Thus, it is assumed that the procedures used in this step may reduce contamination of the vegetable oil (Bordin et al., 2014). Aflatoxins decompose at high temperatures ranging from 237 to 306°C. Thus, the conditions adopted in deodorizing process can be essential for complete removal of aflatoxins from vegetable oils (Bordin et al., 2014).

Parker and Melnick (1966) were the first researchers to assert that the refining process is effective in removing aflatoxins. The authors evaluated the effect of chemical refining on peanut crude oil initially infected with 812 μg/kg aflatoxin, being possible after the bleaching stage, obtaining an oil containing traces of aflatoxins in a concentration lower than 1 μg/kg.

Banu and Muthumary (2010) reported that among the 23 different crude sunflower oil samples were tested, 10 of them showed positive results to AFB_1 and the remaining 13 showed negative results to AFB_1. All the refined oil samples were free from AFB_1 contamination. This was supported by the absence of fungi in the refined oil samples. This may be due to oil processing which includes continuous neutralization, bleaching and deodorizations. During these processes, fungal propagules are probably removed from the oils. The toxic AFB_1 have been found to be heat stable up to their melting points of around 250°C. Therefore, AFB_1 was not completely destroyed by such processes and was carried along the way from seeds to oil samples. The complete conventional processes remove these compounds from the crude oil. But the quantity of contamination is very least ranging from 0.1 to 0.4 ppm. This low level was due to extraction of oil using food grade hexane. The extraction plays a role of partially removing aflatoxin from the oil samples.

CONCLUSION

Knowledge of contaminating sunflower mycoflora is important because undetectability of a mycotoxin at the time of analysis does not mean that this metabolite could not be found later if the toxigenic species is present in the sunflower, and if favorable conditions allow for fungal development and mycotoxin formation. Control of moisture and temperature levels of these commodities is necessary to prevent mould growth and mycotoxin production.

Mycotoxins are partially destroyed in refined oils by the conditions employed in the refining stages. Regardless the process for obtaining oil by pressing or solvent from a feedstock contaminated with aflatoxins, studies have showed that the toxins are partitioned between the oil and meal, requiring the application of physical, chemical or biological products for the reduction or elimination of these contaminants. There is limited evidence indicating that the process of refining crude oil was efficient for removing not only the aflatoxins, but also other mycotoxins produced by *Fusarium* spp., such as trichothecenes and zearalenone. Thus, it is possible to ensure safe edible oil for human provided it is properly processed. (Bordin et al., 2014).

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APPLICATION OF COLD NEUTRALIZATION IN SUNFLOWER OIL REFINING

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ABSTRACT

The aim of refining oils is to remove impurities from crude oils and to give them edible properties. The crude oils may be subjected to degumming, neutralization, bleaching, deodorization, and possibly winterization. In continue system of chemical refining; neutralisation and degumming processes were done with together at the same stage. Then oil bleached with adsorbents and deodorised. Finally in winterization step, oil is cooled, waxes and stearins crystallized and then filtered with helping agent perlit. Cold neutralization is a new process for chemical refining used in a few oil factories in Turkey. In cold neutralization, a part of the winterization step, neutralization and degumming is carried out together. The necessary amount of phosphoric acid is added to oil, mixed and cooled to 5 ^{o}C. Then required amount of caustic added to oil and kept in the crystallization tank at 5 ^{o}C for 12 hours and then waxes and soap-stock are removed by centrifugation from oil. The % 90 of stearin and waxes of oil is removed in cold neutralization. This application simplifies the winterization step in conventional chemical refining. The amount of perlite used in the winterization step decreases, it becomes easier filtration, the soap stock that contains waxes is sold as a more economical byproduct. In this research, the effects of hot and cold neutralization on oil quality especially waxes and oil loss during neutralization, the capacity usage and labor requirements in cold neutralization have been revealed.

Key words: cold neutralization, sunflower oil, oil refining

COMPARISON OF GAS CHROMATOGRAPHY AND NEAR-INFRARED REFLECTANCE SPECTROSCOPY METHODS FOR THE DETERMINATION OF FATTY ACID COMPOSITION OF SUNFLOWER SEED

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ABSTRACT

This study was performed in order to evaluate performance of Near-Infrared Reflectance Spectroscopy (NIRS) which is used to determine oil and fat content, oleic and linoleic acid percentages of sunflower seed by comparing with soxhelet extraction and gas chromatography methods (GC). Oil and fat contents of 34 different sunflower seeds were determined by soxhelet extraction method. Oleic and linoleic acid contents were then evaluated using GC equipped with capillary column and FID detector. Sample spectrums and approximate results were determined by a previously developed calibration method with determinate parameters (RSQ:0.8001, 0.9960, 0.9974 for raw oil content, oleic acid and linoleic acid, respectively) using Foos NIRS System XDS near-infrared Rapid Content Analyzer. Average oleic and linoleic contents of sunflower seeds using GC equipped with capillary column and FID detector were calculated as 53.24 and 33.13%. When the averages of oleic and linoleic acid contents were determined by NIRS, average oleic acid content were found to be 53.40% while 33. 48% was average linoleic acid content. The results of this study showed that NIRS-based method can be reliably used in the determination of oil and fat content, oleic and linoleic acid contents of sunflower seeds. The important contribution of this study is that NIRS-based method can be used as a quick and accurate method in the marketing of sunflower seed and as an environment-friendly method since no chemicals used in this method.

Keywords: Sunflower oil, Oil and fat content, Oleic acid content, Linoleic acid content, NIRS

INTRODUCTION

History dating back to 3000 B.C. and cultivating in a large area in Turkey, sunflower (Helianthus annuus L.) is considered to be one of the most important oil plants in both in Turkey and in the world with the rate of 22-55% oil content. With approximately six million decares planting area, sunflower can be grown in almost all regions in Turkey and contains high amount and good quality oil (TÜİK, 2016). It supplies half of our vegetable oil consumption (BYSD, 2016).

Sunflower oil containing approximately 15% saturated, 85% unsaturated fatty acid and consisting of 14 - 43% oleic and 44 -75% linoleic acid in its unstaurated fatty acids,standard type sunflower oil is one of the most important vegetable oil in terms of oil composition. It is

also among most important oils in human nutrition. In recent years, a range of variety and quality sunflower oil has been produced via the development of mid-oleic type (43.1-71.8%) and high oleic type (75-90.7%) sunflower varieties that has higher oleic acid content than standard sunflower type. There is a growing need for the oils that are less saturated, resistant to oxidation and durable to heat treatment by the change of our consumption habits. In last years, there is also another growing interest in sunflower oil due to its use in other fields apart from food industry. Since the development of high oleic sunflower hybrids, sunflower oil has become more important raw material for the oleochemical industry which includes cosmetics industry.

Food safety and food quality that have close relations with social development and human health are still considered as an important issue in all countries of the world. Day after day, consumers are searching for quality labels on food products and signs that are reliable, they expect high quality products from manufacturers. All of these factors emphasize the importance of reliable techniques for assessing the quality of food.

When the application needs are taken into consideration, the development of fast and effective methods like NIRS technology become apparent. In recent years, there is a growing interest in fast, reliable and enviromentally friendly technologies both in food production and food research. Consequently alternative technologies are being developed to conventional ones. One of the most important of these technologies widely used is the NIR spectroscopy (Cen and He 2007). Used in food analysis after appropriate calibration, fast, reliable and environmentally friendly, NIR Spectroscopy is a technology used for analysis and based on electromagnetic radiation absorbtion in 400-2500 nm wavelength range. (Davies ve Granth, 1987).

NIR spectroscopy , based on the resolution of the analytical and quality factors from food samples with correleation of electromagnetic absorption at aforementioned wavelength, allows to be used routinely in sensory, physical and chemical analysis of food and agricultural products. (Williams ve Norris 1987). For this purpose, studies were conducted to determine crude fat content of the oil plant and fatty acid composition by NIR spectroscopy. (Velasco ve ark. 2004; Koprna ve ark. 2006; Akkaya ve ark. 2015).

It is necessary to determine oil content and fatty acid composition by fast and reliable methods in sunflower seed which holds an important place both in national production and in importation. This study was performed in order to evaluate performance of NIR Spectroscopy which is used to determine oil and fat content, oleic and linoleic acid percentages of sunflower seed by comparing with soxhelet extraction and gas chromatography methods (GC).

MATERIALS AND METHODS

In this study, 34 pieces of different sunflower varieties in which reclamation and adaptation studies are done in East Mediterranean Agronomic Institute testing ground are used as material. Crude oil ratios were determined by Soxhlet extraction method, the ratio of oleic acid and linoleic acid was determined by FID detector gas chromatography capillary column method of sunflower seed oil samples.

FOSS NIRSystem XDS near-infrared Rapid Content Analyser apparatus is used to receive spectrums and determine estimated values of the spectrum of sunflower samples in which classical analysis were completed . Spectra of ground sunflower seed samples were taken to be every 2 nm in between 400-2500 nm wavelength. In determining the estimated

value, the information belongs to NIRS analysis calibration model which is previously developed by using WinISI III v1.61 software package, can be seen in Table 1.

Table 1 The statistics belong to calibration method developed to estimate dry matter, crude oil,oleic acid and linoleic acid rates

Properties	Average±SD	Min (%)	Max (%)	RSQ	SEP	Bias	Slope
Dry Matter Rate (%)	93.79±0.94	90.95	96.62	0.9026	0.286	0.000	1.000
Crude Oil Rate (%)	36.31±5.52	19.75	52.87	0.8001	2.394	-0.076	0.991
Oleic Acid Rate (%)	46.87±16.15	0	95.31	0.9960	0.946	0.043	0.998
Linoleic Acid Rate (%)	40.64±15.04	0	85.75	0.9974	0.793	-0.054	0.998

SD: Standart Deviation, RSQ (Coefficient of determination of Calibration), SEP (Standart Eror of Prediction)

RESULTS

The estimated values of the research in NIRS analysis device and the values determined by conventional analysis methods can be seen in Table 2. In this study, crude oil contents were determined between %32.80 and %48.40 by soxhelet oil extraction method, oleic acid ratio were determined between %34.41 and %80.29 by FID detector capillary column gas chromatography method while the ratio of linoleic acid was determined between %6.45 and %51.93 . Crude oil ratio estimated at NIRS is between 32.45% and 49.61%, oleic acid ratio is between 32.03% and 87.77%, linoleic acid ration is ranged from 3.66% to 51.29% . The average value of the crude oil determined by Soxhlet extraction method was %40.06 while the average values of crude oil estimated by NIRS was found to be %40.44. The average values of oleic acid determined by FID detector gas chromatography capillary column method was %53.24, the average value of linoleic acid was %33.13, while the average value estimated by NIRS was 53.40 for oleic acid and 33.48 for linoleic acid.

In conclusion, this study demonstrates that NIRS can be used reliably to determine crude oil, oleic acid and linoleic acid rates in sunflower seeds. In addition, it shows that NIRS analysis method can be fast and effective analysis method in both vegetable oil industry and sunflower seed trade and marketing and be greener compared to conventional chemical analysis methods due to fact that it has no use of any chemicals.

Table 2. Average values were determined by conventional analysis and average values were estimated by NIRS in sunflower seed samples

Sample No	Conventional Analysis Crude Oil Rate (%)	NIRS Crude Oil Rate (%)	Conventional Analysis Oleic Acid Rate (%)	NIRS Oleic Acid Rate (%)	Conventional Analysis Linoleic Acid Rate (%)	NIRS Linoleic Acid Rate (%)
1	44,93	45,80	60,64	64,30	25,88	22,84
2	40,96	38,86	43,12	40,40	44,24	45,06
3	41,43	41,19	44,56	42,83	40,14	44,09
4	43,68	42,18	46,61	49,13	40,79	39,78
5	33,01	32,45	75,03	70,02	12,24	14,56
6	32,80	33,62	80,09	75,89	7,11	10,20
7	40,73	41,08	34,41	32,03	51,93	51,29
8	41,00	42,63	78,53	78,66	7,72	7,00
9	46,96	47,23	45,01	43,17	42,35	42,49
10	39,11	39,13	55,3	53,97	31,65	30,70
11	44,47	43,76	80,29	87,77	6,45	3,66
12	34,22	34,93	46,47	43,25	36,31	40,78
13	34,52	33,71	49,78	49,84	36,11	34,73
14	47,53	48,23	45,06	45,92	41,01	40,39
15	40,11	41,16	47,04	46,41	40,43	42,48
16	42,67	41,10	58,14	61,41	23,74	25,15
17	43,15	40,57	49,85	54,37	36,91	34,52
18	42,20	43,97	48,24	44,76	37,81	39,34
19	37,16	39,25	52,55	58,27	33,14	31,38
20	48,40	49,61	43,67	44,43	44,56	44,31
21	38,32	38,25	43,36	48,00	42,79	42,43
22	41,65	42,80	51,79	51,62	34,78	33,94
23	43,67	44,72	46,2	45,63	42,51	43,39
24	23,57	33,48	44,32	44,86	40,2	39,41
25	40,83	41,60	48,04	45,94	39,6	42,77
26	47,26	47,61	50,88	54,99	35,69	34,86
27	39,05	36,80	52,27	49,88	32,86	36,08
28	33,81	34,99	43,19	41,40	43,72	42,68
29	43,25	44,14	44,08	40,15	40,46	45,19
30	39,81	38,86	56,28	59,38	28,39	28,52
31	39,17	39,64	50,58	54,86	36,18	33,62
32	33,86	33,64	61,86	59,02	26,42	27,53
33	43,79	44,64	55,06	59,40	32,96	28,98
34	35,07	33,27	77,87	73,57	9,36	14,29
Max Value	48,4	49,61	80,29	87,77	51,93	51,29
Min Value	32,8	32,45	34,41	32,03	6,45	3,66
Average	40,06	40,44	53,24	53,40	33,13	33,48

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AROMA DETERMINATION OF A REFINED SUNFLOWER SEED OIL BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY USING DIFFERENT EXTRACTION METHODS

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ABSTRACT

The sunflower (*Helianthus annuus* L.) seeds are eaten raw, roasted, cooked, dried, and ground, and used as a source of oil. Edible vegetable oils are important to our daily life by providing energies, nutritional compounds, and desirable flavors. Sunflower seeds are usually processed in large oil mills using solvent to extract oil and refining it. Three typologies of sunflower oil, characterized by diverse percentage of oleic acid are present on the market: a low, mid and high oleic sunflower oil. Refined sunflower oil, especially high-oleic, is very versatile and due to its neutral flavour and heat stability it can be consumed in many ways in the kitchen, such as frying and cooking. Edible oils play a significant role in the food industry due to both their functional and nutritional features and their impact on taste, aroma and health. Aroma is a main quality factor for edible vegetable oils as a characteristic parameter. Many extraction techniques have been carried out to extract the aroma compounds of oil. Therefore, in this study, aroma compounds of a refined sunflower oil obtained from a local market in Adana was extracted by different isolation methods including solid phase extraction (SPE), simultaneous distillation extraction (SDE) and purge and trap extraction (PTE). Afterwards, aroma compounds of the extracts were identified and quantified by gas chromatography (GC) coupled with a mass spectrometry (MS) and flame ionization detector (FID). Among the extraction methods, the PTE was quantitatively and qualitatively detected as the most suitable method for the extraction of aroma compounds in the studied sample.

Keywords: Refined sunflower oil, aroma profile, extraction techniques, GC-MS

THE EFFECT OF THE ESSENTIAL OIL FROM *CITRUS AURANTIUM* AS A SOURCE OF NATURAL ANTIOXIDANT IN SUNFLOWER OIL

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ABSTRACT

Edible vegetable oils undergo the oxidation, e.g. oxygen in the air during storage or heat process and etc. As a result of the oxidation, undesirable rancid taste, changes in colour, losses of odour and flavour, deterioration of essential fatty acids and vitamins occurs in oil. In the manufacturing, oxidation occurs spontaneously in oils because of the physical and technological methods. The synthetic antioxidants like butylated hydroxyanisole (BHA) , butylated hydroxytoluene (BHT) and tert-butylhydroquinone (TBHQ) have been popularly used as the antioxidants in oils; however these chemicals show some undesirable effects on health. The aim of the present study was to determine the effect of the essential oil from C*itrus aurantium (*bitter orange) as a natural source of antioxidant, which is an alternative to widely used and known synthetic antioxidants in the sunflower oil. Different concentrations (0, 200, 400, 600, 800 and 1000 ppm) of the essential oil and BHT (200 ppm) were added to sunflower oil emulsion in uncapped vials and then incubated in darkness for 7 days at 60^0C. Samples were examined at 24 h intervals. The oxidative stability of the samples was evaluated by peroxide value (PV) and free faty acid (FFA). ANOVA results showed that the peroxide value and acidity of the oils in treated with essential oil of C. *aurantium* at the following concentration of 200, 400, 600, 800, 1000 ppm and BHT were significantly lower than those of the control groups. Nevertheless, peroxide and acidity values of these samples increased with increasing time.

Key words: Sunflower Oil, Essential Oil, *Citrus aurantium,* Natural Antioxidant, BHT

INTRODUCTION

Vegetable oils, are very important components of our diet, which undergo oxidation during storage and heating process because of many factors especially oxygen in the air. In vegetable oils, oxidation results in many undesirable consequences such as rancid taste and odours, reduction in the shelf life, decrease the nutritional quality (Sikwese and Doudu, 2007). Therefore, manufacturers prefer to utilize the antioxidants in order to prevent the oxidation.

According to the Turkish Standards, there has been some limitations for the uses of synthetic antioxidants in the oil. It has been previously reported that syntetic antioxidants such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT) and tert-butylhydroquinone (TBHQ) may cause many healthy risks, including cancer and carcinogenesis (Iqbal and Bhanger, 2007). Therefore, using natural antioxidants instead of synthetic ones has become popular in recent years., Today, essential oils are already

commercially available. Some of them are classed as generally recognized as safe (GRAS) food additives in the USA (Burt, 2004).

In recent years, uses of the plant extracts have received ongoing interest on the stabilization of the edible oils. For example, the pomegranate peels extract was found to be a potent antioxidant for the stabilization of sunflower oil (Iqbal et al., 2008); methanolic orange peel extract was reported more superior than that of BHT on the stability of crude peanut oil stored for twelve months at room temperature against oxidative rancidity (Arawande and Borokini, 2015). The essential oils of some medicinal and aromatic plants e.g. thyme, clove, orange peel, coriander, garlic and cumin have been tested for their antioxidant potential in different edible vegetable oils.

The aim of the study was to evaluate the antioxidative effects of the essential oil from bitter orange pell during the storage of sunflower oil. The hydrodistillated essential oil from the pell of *C. aurantium* at different concentrations ranging from 200 to 1000 ppm was tested in the sunflower oil. All treatments were stored at 60^0C during one week. The peroxide value and free fatty acid were analysed on each day. The results were compared with the synthetic antioxidant (200 ppm BHT) and that of the control groups.

MATERIALS AND METHODS

Preparing Essential Oil

The essential oil of the peel of *Citrus aurantium* was hydrodistillated for 3 h using Clevenger type apparatus. After distillation, essential oil was dried with anhydrous sodyum sulphate to remove the water from the distillate and then preserved in dark vials at +4°C for further analyses.

Addition of Additives to Sunflower Oil

The essential oil of the bitter orange peel at various concentrations ranging from 200 to 1000 ppm were separately added to sunflower oil in glass bottles and they were thoroughly shaken for proper mixing. Sunflower oil containing 200 ppm BHT and the one that had including no additive (also described as 0 ppm as the control groups) were also setup. Each glass bottle was appropriately labeled and stored in an open place at $60\ ^0$C.

Testing the Oxidative Stability

The stability of emulsions to oxidation was evaluated each 24 h over a 7-day period by analyzing the peroxide values (PVs) and free faty acid (FFA) levels.

PVs were measured on a daily basis. For this purpose, 2 g of oil was initially weigthed and then dissolved in chloroform (10 ml) and glacial acetic acid (15 ml). This was followed by adding 1 ml of saturated KI solution. The solution was throughly mixed for 1 min and then kept in the dark for 5 min. After addition of distilled water (75 ml), the mixture was titrated against sodium thiosulphate (0.01 N) using starch as an indicator. A blank titration was done parallel to treatment and PVs (meq of oxygen/kg) was calculated using the following formula:

$$\text{Peroxide value} = 1000\frac{SXN}{W}$$

In this formula, S is the volume of sodium thiosulphate solution (blank corrected) in ml; N is the normality of sodium thiosulphate solution (0.01 N) and W is the weight of the oil sample (g) (Anon., 1975).

FFA of each oil sample was monitored each day using the standard method for 7 days (Anon., 2003). For this purpose, a known weight of oil sample (3 g) was dissolved in 95% ethanol (75 ml). The mixture was titrated against KOH (0.01 N) using phenolphthalein as an indicator. A blank titration was done parallel to treatment and FFA (%) was calculated using the following formula:

$$\text{FFA} = \frac{SXNX28{,}2}{W}$$

In this formula, S is the volume of KOH in ml; N is the normality of KOH solution (0.01 N) and W is the weight of oil sample (g).

Statistical Analysis

One-way analysis of variance (one-way ANOVA) was carried out on the results. Data was processed using SPSS for Windows 18.0.0 (SPSS Inc., Chicago, USA).

RESULTS AND DISCUSSION

Figure I. depicts peroxide value (PVs) of sunflower oil stored with the essential oil from bitter orange peel and butylatedhydroxytoluene (BHT) for 7 days. It was observed that sunflower oil containing 200 ppm to 1000 ppm essential oil and 200 ppm BHT had lower peroxide values than those of the control groups during storage.

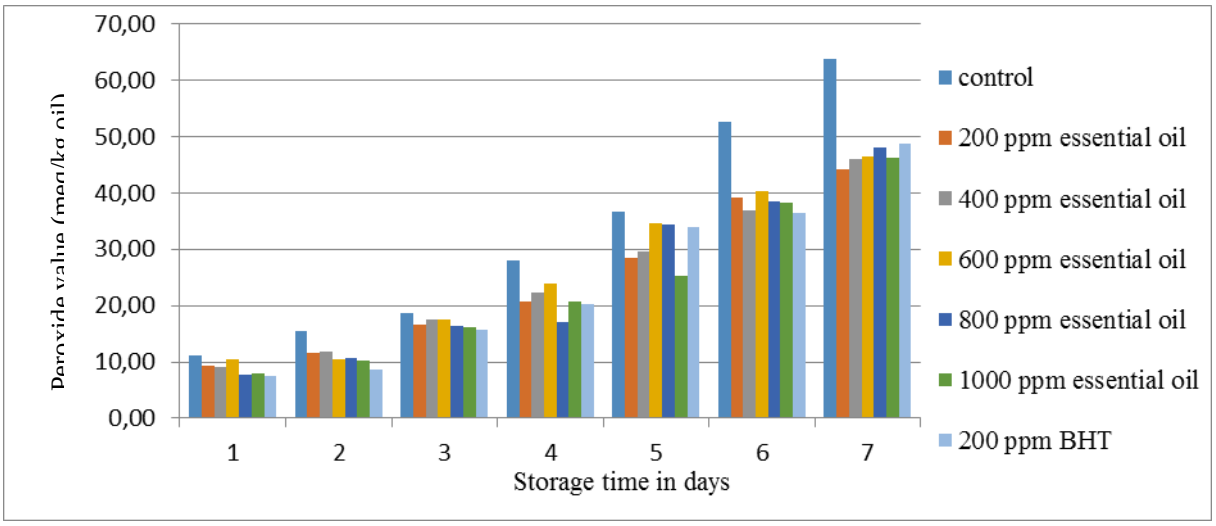

Fig. 1. Peroxide value (meq O_2/kg) of sunflower oil stored at 60^0C

This is in accordance with results of Kamkar et al. (2010) who reported that methanol and water extracts of Iranian pennyroyal in sunflower oil have better antioxidant activities than those of the control groups.

In a study of Shyamala et al. (2005), peroxide value of *M. pulegium* extracts was lowered than the control groups. The present values are in close agreement with findings of Shyamala et al. (2005) who found that extracts of four leafy vegetables which were added to refined sunflower oil conferred a protective effect on peroxide formation.

Figure II. depicts free fatty acid (FFA) of sunflower oil stored with bitter orange peel's essential oil and butylatedhydroxytoluene (BHT) under storage at 60°C for 7 days. It was observed that sunflower oil containing 200 ppm to 1000 ppm essential oil had lower FFA values than control groups during storage.

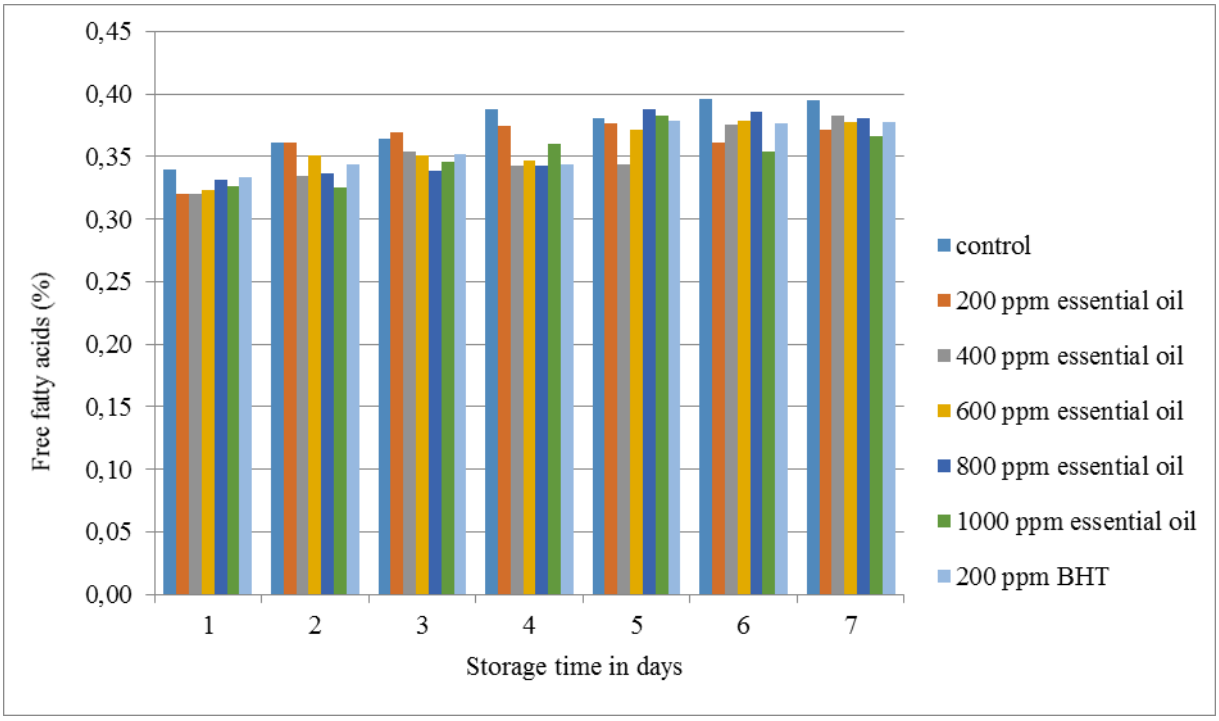

Fig. 2. Free fatty acid content (FFA) of sunflower oil stored at 60^{0}C

These results are in accordance with results of Arawande et al. (2014) who reported that the FFA of oil containing orange peel extract and 200 ppm of BHT was lower than the control groups.

Rehman (2006) also reported that after 6 months of storage, corn oil containing citrus peel extract showed lower FFA contents, and peroxide value levels than the control.

The present results show that all concentrations of the essential oil of *C. aurantium* showed more oxidative stability than that of the control groups. Furthermore, there were no significant differences among the oil groups which include essential oil ranging from 200 to 1000 ppm.

CONCLUSION

The results of the present study apparently indicated that essential oil distillated from bitter orange peels had significant antioxidant activity. It has been widely accepted that the stabilization of the sunflower oil is very difficult because of its high content of linoleic acid.

Two fold concentrations of the essential oil ranging from 200 to 1000 ppm were shown to be strong protective effects against lipid oxidation in the sunflower oil during the storage period. The findings of this study indicated that bitter orange peel exract could be suggested as a potential antioxidant for the stabilization of sunflower oil.

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CHARACTERIZATION OF SUNFLOWER OIL OLEOGELS PREPARED WITH BEESWAX AND SUNFLOWER WAX

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ABSTRACT

In this research we used the sunflower oil to create oleogels with beeswax and sunflower wax as the organogelators at 5% (w/w) addition level. The oleogels were prepared at 90 ºC isothermal conditions, and formed at room temperature overnight. Thermal behaviours of the oleogels were investigated using differential scanning calorimeter (DSC). The sunflower oleogel melting temperature was found to be between 60-65 ºC, while the beeswax oleogel were 45-50 ºC. The crystallization temperatures of the sunflower wax oleogel were ranged from 55 to 60 ºC and beeswax oleogel were from 35 to 40 ºC. The firmness values of oleogels determined by TA-XT Texture Analyzer. The firmness values of the beeswax and sunflower wax oleogels were found around 1.0- 2.0 N and 3.0 – 4.0 N, respectively. These parameters provide information about oleogels hardness and spreadability. The oil binding capacities of beeswax and sunflower wax gels were ≥ 99%. Solid fat contents of beeswax and sunflower wax oleogels determined at 35 ºC were ranged between 2.00 and 2.50%, and 3.00 and 3.50%, respectively. The X-ray diffraction peaks observed at 4.10 and 3.70 Å demonstrated that the oleogels had crystalline structure similar to β′ polymorphs of triglycerides. In conclusion *trans*-free spreads or margarines based on sunflower oil oleogels could be created as solid fat stock alternatives. Sunflower oil could be a valuable alternative to create oleogel stocks to produce margarine, spread, shortening and similar products.

Keywords: Sunflower oil, oleogels, hardness, melting point, X-ray diffraction

QUALITY CHARACTERISTICS OF THE OILS OBTAINED BY COLD PRESSING TECHNIQUE

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ABSTRACT

Cold pressing is used to extract oil from plant seed instead of conventional solvent extraction method since cold-pressing does not require the use of organic solvent or heat. The cold pressing procedure involves neither heat nor chemical treatments, and it is becoming an interesting substitute for conventional practices because of consumers' desire for natural and safe food products. Cold pressing also involves no refining process and may contain a higher level of lipophilic phytochemicals including natural antioxidants. Cold-pressed oils refer to oils that are extracted by cold-pressing plant seed with a screw press or hydraulic press. Cold-pressing is able to retain bioactive compounds such as essential fatty acids, phenolics, flavonoids and tocopherol in the oils. Hence, cold-pressed seed oils contain these bioactive compounds that exert health benefits. Sunflower (*Helianthus annuus*) and canola (*Brassica napus*) seed oils are examples of oils that are extracted by cold-pressing. Cold-pressed oils are considered as healthy oils that are important to human nutrition due to their favorable polyunsaturated fatty acid content, notably α-linolenic acid (C18:3; *n*-3) and linoleic acid (C18:2; *n*-2). Cold pressed oils are a good source of beneficial components, such as antioxidative phenolic compounds and other health-beneficial phytochemicals. Moreover, they are free of chemical contamination. The cold press which not exceed 50 °C preserves bioactive components, such as vitamins, provitamins, phytosterols, phospholipids and squalene). In addition, it has been proven that these components have a positive effect on human health.

Key words: cold press technology, quality characteristics, fatty acids.

EFFECTS OF TEMPERATURE AND VACUUM PARAMETERS APPLIED DURING DEODORIZATION STEP ON SUNFLOWER OIL QUALITY

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ABSTRACT

Deodorization is a crucial refining stage which aims to vaporize odoriferous compounds and free fatty acids from sunflower and other vegetable oils by applying high temperatures and low pressures. Deodorization, the common process used for the refining of edible fats and oils, has an important effect on the quality of refined sunflower oil. Although deodorization targets to remove only undesirable compounds, other components with comparable volatilities are also lost. Deodorization represents a critical step in the refining process as it involves high temperature (180–220 °C) that could induce degradation reactions under low pressure (1–10 mbar). The effect of deodorization temperature has been evaluated on the formation of *trans* fatty acids and long-chain PUFAs (LC-PUFAs) geometrical isomers that influence the final quality of vegetable oils. The main factor that controls the speed of the isomerization reaction is the deodorization temperature. Moreover, long time exposure of high temperature in deodorization enables carotene to be destroyed therefore the process needs to be controlled to minimize carotene decomposition. Distillation under vacuum is a principal process in deodorization. The purpose of this process is the removal of undesired volatile odoriferous components in sunflower and other vegetable oils, namely aldehydes, ketones, and free fatty acids. In conclusion, temperature and vacuum parameters should be under controlled during the deodorization process.

Keywords: deodorization, temperature, vaccum, oil quality

DIFFERENT EXTRACTION METHODS FOR SUNFLOWER AND OTHER EDIBLE OILS

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ABSTRACT

Nowadays, solvent extraction techniques are widely used in the production of sunflower and other vegetable oils. In recent years, increased sensitivity to the environment, efforts to improve the oil yield and quality, the desire to reduce the necessary of refining and to obtain more healthy edible oils are caused new extraction techniques development. These techniques can be considered as supercritical fluid extraction, ultrasound and ultrasound-enzyme assisted extraction, ultrasound-microwave assisted extraction, aqueous enzymatic extraction. Furthermore, these methods are also integrated with the solvent extraction process for sunflower and other edible oil production and applicability was investigated. In this review, the application of these methods in vegetable oil production were discussed.

Key words: ultrasound extraction, supercritical fluid extraction, aqueous enzymatic extraction, oil extraction.

FRYING PERFORMANCE OF HIGH OLEIC SUNFLOWER OILS

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ABSTRACT

Deep fat frying is one of the oldest and popular culinary techniques used in food cooking throughout the world. During the frying process, undesirable reactions like oxidation, hydrolysis, polymerization, isomerization etc. occur and these reactions cause quality deterioration in both oil and fried foods. The frying conditions as the temperature and time, ratio of food to oil, presence of oxygen and metals, and especially the composition of the frying oil used are the main factors that affect the deterioration of frying oil. Conventional sunflower oil is highly susceptible to lipid oxidation because it has approximately 70% linoleic acid content. High oleic (75-90%) sunflower oils have been shown to exhibit significantly higher oxidative stability than regular sunflower oil during frying. In addition, high oleic sunflower oil has the best characteristics (lower conjugated diene, total polar material and free faty acid content) and superior stability compared to the conventional sunflower oil and other commercial oils (soybean, corn, peanut oil etc.) at frying conditions. It was reported that high oleic sunflower oils could improve the health benefits as decreasing in the risk of coronary heart disease and plasma levels of LDL cholesterol susceptibility to oxidation. Hence, high oleic sunflower oils could be considered as a more suitable and cheaper alternative oil for catering and frying industries.

Keywords: Sunflower oil, high oleic, linoleic, frying, stability

COMPARISON OF PHYSICAL AND CHEMICAL PROPERTIES OF SUNFLOWER AND DIFFERENT VEGETABLE OILS BIODIESEL

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ABSTRACT

Biodiesel refers to a vegetable oil or animal fat based diesel fuel consisting of long chain alkyl (methyl, ethyl, or propyl) esters. It can fulfill energy security needs without sacrificing engine's operational performance. In this study, the relationship between the chemical structure and physical properties of biodiesel esters is analyzed and compared with sunflower oil and some vegetable oil diesel. The compared biodiesels were made using sunflower oil and different oils with sodium hydroxide as catalyst. The physico-chemical properties assessed includes, density, flash point, kinematic viscosity, sulfated ash, iodine value.

Key words: sunflower oil; density; flash point; kinematic viscosity

LC-DAD/ESI-MS/MS CHARACTERIZATION OF PHENOLIC COMPOUNDS OF SUNFLOWER OIL

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ABSTRACT

The study investigates the phenolic contents and antioxidant potential of sunflower oils from commercial markets in Turkey were extracted with methanol/water. Extracts were used for the phenolic and antioxidant studies. A simple and reproducible method for qualitative and quantitative analysis of phenolic compounds in sunflower oils, high performance liquid chromatography with diode array detector (HPLC-DAD), and HPLC-mass spectrometry (MS) in tandem mode was developed. Detection and quantification were performed at 280, 320 and 360 nm. For identification purposes, HPLC-MS/MS was equipped with electrospray ion source in the negative and positive-ion mode. Most of the compounds detected were mainly hydroxycinnamic acids. Chlorogenic acid was found as the major compound in the group of phenolic acids followed by vanillic acid, while rutin was determined as the most abundant compound in the overall phenolics of sunflower oil. Rutin has an average concentration of 2.70 mg/kg oil whereas chlorogenic acid has an amount of 1.66 mg/kg oil as the second most dominant phenolic compound. Antioxidant activities of sunflower oils were measured as a comparison of two methods; the DPPH (2,2-diphenyl-1-picrylhydrazyl) and ABTS (2,2′-azino-bis- (3-ethyl-benzothiazoline-6-sulphonic acid) assays. Our results showed strong correlations between antioxidative capacity and total phenolic content of sunflower oils.

Keywords: Sunflower oil phenolics, phenolic characterization, LC-MS/MS analysis, antioxidant assays.

INTRODUCTION

Sunflower (*Helianthus annuus* L.) is a crucial crop producing annually and native to North America (Amakura *et al.,* 2013). It is widely used in oil production and sunflower oil ranks fourth in world vegetable oil production, after palm oil, soybean oil and canola oil (Weisz *et*

al., 2009). Sunflower oil is also popularly used in Turkey and the seed production in 2013 was 1523000 tons (FAO-STAT,2013). Sunflowers date back to 26th century, are known and used till then (Pope *et al*., 2001).Sunflower oil (sunflower seed oil) is an abundant source of unsaturated fat, vitamin E, and some phenolic compounds (Fiori, 2009). Sunflower seeds are affluent in oil and with this oil having high ratio of polyunsaturated/saturated fatty acids and high linoleic acid content, sunflower oil is considered to be good for human consumption (Salgın *et al*., 2006).

Sunflower seeds have been shown to have antioxidant activities and are a very good source of vitamin E and several B-vitamins. Moreover, sunflower seeds contain a number of phenolic compounds largely responsible for the modifications occurring during the processing of sunflower seeds (Weisz *et al*., 2009). Phenolic compounds have been proposed to be the potent and important contributors in reducing oxidative stress due to their antioxidant activity, which are of great importance. Therefore, food industry is concentrating on foods containing various bioactive compounds for health promotion and disease prevention (Kelebek *et al*., 2015a).The major phenolic constituents of sunflower seeds are chlorogenic acid, smaller quantities of caffeic acid, cinnamic, coumaric, ferulic, sinapic and hydroxy-cinnamic and finally traces of vanillic, syringic and hydroxy-benzoic acids (Pedrosa *et al.,* 2000), but the phenols are present only in traces in sunflower seed oils due to the oil production process (Leung *et al.*, 1981).

In this research, the determination of phenolic content and their antioxidant activity was aimed. As regarding the lack of studies in these terms of sunflower oil, this paper will be helpful in understanding the characterization of sunflower oils.

MATERIAL AND METHODS

Chemicals

Methanol, acetonitrile, formic acid, and cyclohexane HPLC-grade solvents were purchased from Riedel-deHaen (Switzerland). All other reagents used were of analytical grade. Ultrapure water generated by the MilliQ system (Millipore, Bedford, MA) was used. Phenolic compounds (p-hydroxybenzoic, vanillic acid, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, sinapic acid, qurcetin-3-galactoside, kaempferol-3-glucoside and rutin) were obtained from Sigma-Aldrich (Steinheim, Germany).

Samples

Sunflower oil samples were collected from domestic markets in Adana, Turkey. 5 different commercial brands of oils were analysed.

Extraction of the Phenolic Fraction

According to Rotondi *et al*. (2004), 4 g of the oil sample was added to 2 mL of n-hexane and 4 mL of a methanol/water (70/30; v/v) solution in a 10 mL centrifuge tube. After vigorous mixing, they were centrifuged for 15 min at 5500 rpm. The hydro-alcoholic phase was collected, and the hexane phase was re-extracted twice with 2 mL of methanol/water (70/30; v/v) solution each time. Finally, the hydro-alcoholic fractions were combined, washed with 2 mL of n-hexane to remove the residual oil, then concentrated and evaporated in vacuum at 35 °C. The dry extracts were re-suspended in 0.5 mL of a methanol/water (50:50, v/v) solution and filtered through a 0.2 μm nylon filter (Whatman Inc., Clifton, NJ) before being analyzed by LC-ESI-DAD-MS/MS.

LC-DAD-ESI-MS/MS analysis of phenolic compounds

An Agilent 1100 HPLC system (Agilent Technologies, Palo Alto, California, USA) operated by Windows NT-based ChemStation software was utilized; the HPLC equipment was used along with a diode array detector (DAD). The system comprised a binary pump, degasser, and auto sampler. The column used was a Phenomenex reversed-phase C-18 column (4.6 mm × 250 mm, 5 μm) (Torrance, California, USA). The mobile phase consisted of two solvents: Solvent A, water/formic acid (99.5:0.5; v/v) and Solvent B, acetonitrile/solvent A (60:40; v/v). Phenolic compounds were eluted under the following conditions: 0.5 ml min^{-1} flow rate with temperature set at 25 °C; isocratic conditions from 0 to 5 min with 0% B; gradient conditions from 0% to 5% B in 20 min; from 5% to 15% B in 18 min; from 15% to 25% B in 14 min; from 25% to 50% B in 31 min; from 50% to 100% B in 3 min; followed by washing and reconditioning of the column. The ultra-violet-visible spectra (scanning from 200 nm to 600 nm) were recorded for all peaks. Triplicate analyses were performed for each sample. The identification and assignation of each compound was performed by comparing retention times and UV spectra to authentic standards; and confirmed by an Agilent 6430 LC-MS/MS spectrometer equipped with an electrospray ionization source. The electrospray ionization mass spectrometry detection was performed in negative ion mode with the following optimized parameters: capillary temperature 400°C, N_2 12 L/min; nebulizer pressure, 45 psi (Kelebek *et al*., 2015a). Data gaining was performed using the Multiple Reactions Monitoring (MRM) method that solely monitors specific mass transitions during preset retention times. The curves were obtained using the commercial standards of the concentrations normally present in sunflower oils (approximately 1-100 mg kg^{-1}), obtaining regression coefficients (r^2) above 0.995 in all cases.

Measurement of antioxidant activity

DPPH Assay: 0.1 mL of diluted sunflower oil extract was mixed with 3.9 mL of DPPH solution (2.36 mg/100 mL methanol) and vigorously vortexed. The solution was held in the dark at ambient conditions for 15 min. The absorbance was measured at 517 nm by a UV-Visible spectrophotometer (Shimadzu UV-1201, Kyoto-Japan). Trolox calibration curve was used to calculate the antioxidant activity of the oil extracts and to express the antioxidant capacity in mM Trolox equivalent per kg of sunflower oil. The mean and standard deviation were calculated for the three replicates (Kelebek *et al*., 2015a; Kesen *et al*., 2013).

ABTS Assay: The ABTS solution was created at a concentration of 7 mM and mixed with 2.5 mM of potassium persulphate, and stored after incubation at 23 °C in the dark for 12–16 h. The ready-made solution was diluted with 80 % methanol to measure an absorbance of 0.7±0.01 at 734 nm. Then, 3.9 mL of ABTS solution was added to 0.1 mL of the oil samples and mixed vigorously. Finally 10 min. were waited to ensure reaction and the absorbance was monitored at 734 nm [13, 14]. The calibration curve equations related to the Trolox standard were y=0.0004x + 0.0089 with R2= 0.9996 for ABTS and y=0.0004x + 0.0082 with R2= 0.9995 for DPPH within a concentration range from 5 to 150 μmol/L.

RESULTS AND DISCUSSION

Phenolic Compounds of Sunflower Oil

Table 1 lists the compounds identified according to different families, including the information provided by HPLC-DAD-ESI-MS/MS analysis: retention time, λ_{max} in the ultraviolet region, molecular ion, main fragment ions in MS/MS, and tentative identification. A total of 10 phenolic compounds were identified and quantified in oils, including p-hydroxybenzoic acid, vanillic acid, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, sinapic acid, quercetin-3-galactoside, kaempferol-3-glucoside and rutin. Rutin is the

major phenolic compound in sunflower oils followed by chlorogenic acid as they constitute the large proportion of the total phenolic content (Table 2). These two compounds were reported to present in the seed and kernel of the sunflowers in addition to caffeic and ferulic acid with chlorogenic acid being the most abundant compound that is one of the natural phenols includes one molecule of caffeic acid and one of quinic acid. Caffeic acid is reported to found more in the kernels of the sunflower than the chlorogenic acid Žilić *et al*., 2010. Phenolic acids have higher proportion in phenolic compounds of sunflower oils. Rutin is a flavonol formed by a quercetin which is a flavonol and a rutinoside, a dissaccharide (Kelebek *et al*., 2015b). This compoundhas the majority having a concentration between 2.23 and 2.99 mg/kg in flavonoids of sunflower oil Chlorogenic acid has a varying concentration between 1.40 and 1.80 mg/kg followed by vanillic acid (1.14-1.35 mg/kg) in phenolic acids. The other phenolic acids present in traces due to the refining process of the oil (Leung *et al*., 1981). Sunflower oil phenolic acids show similarity with phenolics of olive oil including vanilic, caffeic, ferulic and p-coumaric acid (Godoy-Cabarello *et al*., 2012; Kelebek *et al*., 2012) while olive oil having total phenol content approximately two or three times more than sunflower oil (Guzel *et al*., 2009). Also the amounts of phenolic compounds is known to vary according to the conditions of the region where in the crop grows, the extraction methods and the conditions of storage (Kelebek *et al*., 2012).

Antioxidant Activity of Sunflower Oil

Antioxidant capacity was measured by two methods namely, ABTS and DPPH assays. Table 3 presents the results of the antioxidant activities obtained by the sunflower oils. As it can be seen from the results, ABTS assay stated better the antioxidant activity of phenolic compounds than the DPPH assay as the method gave higher values.

DPPH is a free radical scavenging method, being simple, rapid and repeatable, preferably used in determining the antioxidant activity of compounds (Kelebek *et al*., 2015b). On the other hand, ABTS is used more in the food and agriculture industry which is clearly the better method for evaluating the antioxidant capacity of sunflower oils (Kelebek and Selli, 2011). Antioxidant capacities were found as 7.16 μM Trolox/kg oil using DPPH assay and 11.76 μM Trolox/kg oil by ABTS assay in average while the maximum values were 7.71 and 12.76 respectively. Rutin is known to have antioxidant, antiinflamatory activities and can be used in preventing cancer diseases (Kelebek *et al*., 2015b). In addition to the antioxidant behavior, chlorogenic acid shows antiviral, hypoglycaemic and hepatoprotective activities yet caffeic acid is reported to have higher antioxidant capacity (Dixon *et al*., 1995; Chen and Ho, 1997).

CONCLUSION

A total of ten phenolic compounds were isolated from sunflower oil samples and identified by HPLC-DAD-ESI-MS/MS analysis. Rutin is found as the most dominant phenolic compound with a concentration of 2.70 mg/kg oil followed by chlorogenic acid (1.66 mg/kg) and vanillic acid (1.35 mg/kg). The other phenolic acids present in sunflower oil are determined as p-hydroxybenzoic acid, caffeic acid, p-coumaric acid, ferulic acid and sinapic acid while flavonoids include quercetin-3-galactoside and kaempferol-3-glucoside. Antioxidant capacity of these phenolic compounds was determined as a comparison of DPPH and ABTS methods. ABTS assay is found as more appropriate in determining antioxidant activity of sunflower oil. By using DPPH assay antioxidant capacity is determined as 7.16 μM Trolox/kg oil while ABTS had a result of 11.76 μM Trolox/kg oil. In conclusion, the phenolic content and antioxidant activity supply a beneficial contribution to the characterization of sunflower oils. Regarding this matter, further investigation is advised.

Table 1. HPLC-DAD-ESI-MS/MS identification of phenolic compounds

Peak	Compounds	MF	λ (nm)	Fragmentor (v)	Precursorion	Collision energy (v)	Quantitative transition (m/z)
	Phenolic acids (PA)						
1	p-hydroxybenzoic acid	$HOC_6H_4CO_2H$	256	90	137	15	137>93
2	Vanillic acid*	$C_8H_8O_4$	258, 293	90	167	15	167>123
3	Chlorogenic acid*	$C_{16}H_{18}O_9$	326	90	353	15	353>191
4	Caffeic acid*	$C_9H_8O_4$	325	90	179	15	179>135
5	*p*-coumaric acid*	$C_9H_8O_3$	236, 310	90	163	15	163>119
6	Ferulic acid*	$C_{10}H_{10}O_4$	323, 293	90	193	15	193>134
7	Sinapic acid*	$C_{11}H_{12}O_5$	324	90	223	15	223 >149
	Flavonoids (FLA)						
8	Quercetin-3-galactoside*	$C_{21}H_{20}O_{12}$	353	90	463	15	463>301
9	Kaempferol-3-glucoside*	$C_{21}H_{20}O_{11}$	348	90	447	15	447>285
10	Rutin*	$C_{27}H_{30}O_{16}$	360	90	609	15	609>301

Table 2. Phenolic content of sunflower oil extracts (mg/kg oil)

Phenolic Compounds	p-hydroxybenzoic acid	Vanillic acid	Chlorogenic acid	Caffeic acid	p-coumaric acid	Ferulic acid	Sinapic acid	Qurcetin-3-galactoside	Kaempferol-3-glucoside	Rutin	*Total*
Sample 1	0.61±0.01	1.46±0.00	1.80±0.03	1.04±0.01	0.52±0.01	0.38±0.01	0.33±0.00	0.09±0.00	0.13±0.00	2.75±0.04	***9.10±0.10***
Sample 2	0.54±0.01	1.40±0.01	1.72±0.02	0.98±0.01	0.57±0.00	0.44±0.01	0.21±0.01	0.11±0.00	0.15±0.00	2.99±0.01	***9.10±0.13***
Sample 3	0.58±0.01	1.43±0.02	1.76±0.03	1.01±0.02	0.55±0.01	0.41±0.01	0.27±0.01	0.10±0.01	0.14±0.00	2.87±0.03	***9.10±0.10***
Sample 4	0.53±0.02	1.31±0.01	1.62±0.02	0.93±0.01	0.50±0.03	0.37±0.01	0.25±0.02	0.09±0.01	0.13±0.00	2.64±0.02	***8.38±0.10***
Sample 5	0.47±0.01	1.14±0.03	1.40±0.02	0.80±0.01	0.42±0.01	0.31±0.00	0.23±0.01	0.08±0.00	0.11±0.00	2.23±0.01	***7.18±0.08***
Min	***0.47***	***1.14***	***1.40***	***0.80***	***0.42***	***0.31***	***0.21***	***0.08***	***0.11***	***2.23***	***7.18***
Max	***0.61***	***1.46***	***1.80***	***1.04***	***0.57***	***0.44***	***0.33***	***0.11***	***0.15***	***2.99***	***9.10***
Mean	***0.55***	***1.35***	***1.66***	***0.95***	***0.51***	***0.38***	***0.26***	***0.09***	***0.13***	***2.70***	***8.57***

Table 3. Antioxidant capacities of sunflower oil extracts (μM Trolox/kg oil)

	DPPH	**ABTS**
Sample 1	7.05±0.54	11.58±0.88
Sample 2	7.45±0.26	12.23±0.43
Sample 3	7.08±0.96	11.61±1.57
Sample 4	7.71±0.41	12.66±0.68
Sample 5	6.52±0.60	10.70±0.99
Min	6.52±0.26	10.70±0.43
Max	7.71±0.96	12.66±1.57
Mean	7.16±0.56	11.76±0.91

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COMPARISON OF ENZYMATIC PROCESS FOR BIODIESEL PRODUCTION FROM SUNFLOWER OIL

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ABSTRACT

The research on the production of biodiesel has increased significantly in recent years because of the need for an alternative fuel which endows with biodegradability, low toxicity and renewability. In order to design an economically and environmentally sustainable biodiesel production process, a proper understanding of the factors affecting the process and their relative importance is necessary. A comprehensive review of the literature on the subject of biodiesel production was carried out. Traditionally biodiesel has been produced using either acid or base catalysts. The multi-step purification of end products, wastewater treatment and energy demand of the conventional process has led to search for alternative option for production of biodiesel. The use the enzyme lipase as a biocatalyst for the transesterification reaction step in biodiesel production has been extensively investigated. The enzymatic process is known to be a clean and environment friendly technique for biodiesel production. The present review analyzes enzymatic process of some vegetable oils reported in literature and also suggests a suitable method for commercialization of the enzymatic process.

Keywords: biodiesel; enzyamtic process; sunflower oil, vegetable oils

ASSESSMENT OF SUNFLOWER OIL ADULTERATION

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ABSTRACT

The aim of this study is to estimate the sunflower oil adulteration with cheaper oils as raw cottonseed and canola oils. Sunflower oils, raw canola oil and raw cottonseed oil samples were supplied from market to investigate the possibility of adulteration. Main fatty acid composition of samples was detected by using GC-MS. L^*, a^* and b^* color values of the samples were also determined to detect the correlation with fatty acid composition. Increase of linolenic acid and palmitic acid percentages of sunflower oils samples was a good indicator for estimation of canola oil and palm oil addition, respectively. Some of the sunflower oil samples were suspected to be adulterated. L^*, a^* and b^* color values were also discussed on prediction of the possibility of adulteration. b^* values were detected to be higher in suspected oils. Detailed statistical modeling studies may be recommended for estimation of cheaper raw oil addition in sunflower oil.

Key words: Adulteration, Fatty acid, Sunflower oil, Color

INTRODUCTION

Sunflower is botanically classified as *Helianthus annuus* and is an annual plant. It is thought to have been domesticated around 1000 B.C. by Native Americans. People in many regions began to process vegetable oils, from many oil sources for cooking purposes, before thousands of years ago. In 1860, Russia farmers cultivated sunflower. At that time, they became the world's largest producer of sunflower seeds. (Anonymous, 2010).

Sunflower oil is rich in linoleic acid and it is one of the most economically important vegetable oil source, especially in Turkey. Also, the widely usage of cake/meal of sunflower, obtained after oil extraction, as livestock increases the economic value of sunflower (İncekara, 1972, Dayal et al., 2011).

The tendency of adulteration on olive oil is higher in comparison to other oils, so there are many researches on detection of the adulteration in olive oil (Gegiou and Georgouli, 1983; Mannina et al., 1999; Blanch et al., 1998, 1999, 2000; Salivaras et al., 1992; Dionisi et al., 1995; Flor et al., 1993). Sunflower oil is cheaper than many other oils and sometimes used for adulteration of olive oil (Savaş, 1969) but, in recent years sunflower oil is also subjected to be adulterated with some other cheaper oils.

The fatty acid composition of sunflower oil may vary by the effect of many reasons. Republic of Turkey Ministry of Food, Agriculture and Livestock published a regulation on the 12ht April 2012, called as 'Bitki adı ile anılan yağlar tebliği'. The Ministry announced the ranges of fatty acid composition of many vegetable oils (Anonymous, 2012). It is attractive that the lower and the higher limits of the ranges are at their maximum and in accordance with the literature.

Raw canola oil and raw cottonseed oil are cheaper than sunflower oil was subjected to this study for our suspect of their use in adulteration of sunflower oil. The ranges of fatty acid composition of sunflower, canola and cottonseed oil's, mentioned in regulation, are shown in Table 1.

Table 1. The ranges of fatty acid composition of sunflower, canola and cottonseed oil in Turkish Food Codex on vegetable oils (%)*

Fatty acids		Sunflower oil	Canola oil	Cottonseed oil
Caproic	(C6:0)	nd[a]	nd	nd
Caprylic	(C8:0)	nd	nd	nd
Capric	(C10:0)	nd	nd	nd
Lauric	(C12:0)	nd - 0.1	nd	nd – 0.2
Myristic	(C14:0)	nd - 1.0	nd - 0.2	0.6 - 1.0
Palmitic	(C16:0)	4.0 - 7.6	2.5 - 7.0	21.4 – 26.4
Palmitoleic	(C16:1)	nd - 0.3	nd - 0.6	nd – 1.2
Margaric	(C17:0)	nd - 0.2	nd - 0.3	nd - 0.1
Heptadecenoic	(C17:1)	nd - 0.1	nd - 0.3	nd - 0.1
Stearic	(C18:0)	2.1 - 6.5	0.8 - 3.0	2.1 – 3.3
Oleic	C18:1	14.0 - 71.8	51.0 - 70.0	14.7 - 21.7
Linoleic	C18:2	18.7 - 74.0	15.0 - 30.0	46.7 - 58.2
Linolenic	C18:3	nd - 0.5	5.0 - 14.0	nd - 0.4
Arachidic	C20:0	0.1 - 0.5	0.2 - 1.2	0.2 - 0.5
Eicosenoic	C20:1	nd - 0.3	0.1- 4.3	nd - 0.1
Behenic	C22:0	0.3 - 1.5	nd - 0.6	nd - 0.6
Docosahexaenoic	C22:1	nd - 0.3	nd - 2.0	nd - 0.3
Lignoceric	C24:0	nd - 0.5	nd - 0.3	nd - 0.1
Nervonic	C24:1	nd	nd – 0.4	nd

[a]: not detected (≤ % 0,05); *Anonymous, 2012.

The aim of this study is to estimate the sunflower oil adulteration with cheaper oils as raw cottonseed and canola oils by the aid of fatty acid composition.

MATERIALS AND METHODS

Thirtysix sunflower oil, one conola oil and one cottonseed oil samples were obtained from market from many regions of Turkey. Names of companies were hidden.

Color measurement:

Color measurements of the oil samples were carried out using a Minalto CR400 colorimeter. The instrument was standardized each time by a white (L=93.01, a=1.11, b=1.30) tile. The color values were expressed as *L** (whiteness or brightness/darkness), *a** (redness/greenness) and *b** (yellowness/blueness) (Hunter, 1948). 20 ml oil samples were

poured in a petric plate on a white tile for measuring the color values (Morello et al., 2004; Sikorska et al., 2007).

Determination of fatty acid composition:

Fatty acid composition was carried out by Agilent 6890 series GC system (Agilent Technologies, USA) fitted with a capillary column packed with 100% cyanopropyl methyl polysiloxane (Supelco SP-2380 model, 60 m × 250 μm × 0.2 μm i.d.; Bellefonte, PA, USA) and equipped with a flame ionization detector. Before injection, oil samples were converted to fatty acid methyl esters (FAMEs). 0.1 g of oil sample was weighed in a sample tube and dissolved in 10 mL hexane. Then 1 mL of 2 N potassium hydroxide in methanol was added and shaken for one minute before the centrifugation procedure. After centrifugation, the clear supernatant was transferred to a GC auto-sampler vials for injection. One μL FAMEs were injected into the GC-FID system using an auto-sampler with a split ratio of 100:1. The oven's initial temperature was set to 50°C for 2 mins and then increased at a rate of 4°C/min up to 240°C, where it was held for 10 min. Both the injector and the detector temperatures were set to 250°C. The flow rate of carrier gas (hydrogen) and make-up gas (nitrogen) were set to 1 mL/min^{-1} (AOCS, 1984). The data were recorded by using the Agilent ChemStation data processor. FAMEs peaks were identified by comparison with retention times of known standards (Sigma Chemical Co.) and quantification was determined as the percent area of each peak relative to the sum of all peak areas. All analyses were conducted in duplicate and results are provided as average values.

Statistical analysis:

Data were subjected to analysis of variance with mean separation by Duncan's multiple range tests. Differences were considered statistically significant at the $P < 0,05$ level. Statistical analysis was performed using SPSS 10.0 for Windows. The statistical results were evaluated according to Düzgüneş et al., 1987.

RESULTS

The detected *L**, *a** and *b** value ranges for 36 sunflower oil samples were 69,177-70,670, (-1.903) - (-4.233) and 7.597-16.060, respectively. *L** value of the samples were changed in a narrow range but the range for *a** and *b** were wide that reflects the sensitivity on them. *a** value of 30th sample were higher in comparison with other sunflower oil samples. And, the value of *a** was similar to values obtained for cottonseed and canola oils. *b** value was the lowest for 12th sunflower sample and was the highest for the 30th sunflower oil sample (Table 2).

10th and 26th sunflower oil samples were found to be higher in myristic acid content than the other sunflower oil samples as 1,529 % and 4,055 %, respectively. Myristic acid content of the samples doesn't give any confirmative idea on suspicion of adulteration of sunflower oils by the use of canola and cottonseed oil. Other fatty acid profile of these samples was belonging to fatty acid profile of sunflower oil. Especially the detection of high myristic acid content may cause a formation of doubt of adulteration with palm, coconut and babassu oil, but lauric acid was not detected in these samples which may be a parameter for removal of doubt (Table 2).

Table 2. Main fatty acid composition (%) and *L*, a*, b** values of oil samples

Samples	Myrstic acid	Palmitic acid	Oleic acid	Linoleic acid	Linolenic acid	Arachidic acid	Behenic acid	L^*	a^*	b^*
1	0.045 e*	4.924 ö	31.523 ghıijk	62.173 ab	0 c	0.321 k	0.741 efghı	70.090 efgh	-2.720 l	10.613 l
2	0.059 e	6.782 klm	29.675 jklmno	61.371 abc	0 c	0.814 efghıij	1.037 cd	70.460 abcde	-2.757 l	10.790 jk
3	0 g	5.701 mnoö	32.482 fghıi	59.487 bcde	0 c	0.640 hıijk	0.954 cde	70.563 abc	-2.350 f	8.417 u
4	0 g	9.200 ı	28.039 oöp	59.398 bcdef	0 c	1.459 abc	1.535 ab	69.187 m	-2.417 g	8.947 sş
5	0 g	8.375 ıij	34.538 ef	56.430 fghı	0 c	0.334 k	0.380 jk	70.260 defg	-2.870 n	10.873 j
6	0 g	8.304 ıijk	30.959 ıijklm	57.496 efgh	0 c	1.255 bcde	1.628 a	69.693 jkl	-2.507 h	9.840 n
7	0.223 d	8.683 ıi	34.470 ef	55.405 hıi	0 c	0.417 jk	0.646 ghı	70.003 ghıi	-2.677 k	9.530 ö
8	0.044 e	6.368 lmno	29.976 ijklmno	62.506 ab	0 c	0.514 ijk	0.744 efghı	69.910 hıij	-2.940 o	11.193 i
9	0.022 efg	5.656 mnoö	34.957 ef	58.452 cdefg	0 c	0.405 jk	0.748 efghı	70.650 ab	-2.587 ij	9.137 pr
10	1.529 b	7.657 ijkl	40.863 d	48.427 mnoö	0 c	0.348 k	0.660 ghı	70.050 fghı	-2.720 l	9.763 no
11	0.056 e	5.351 oö	44.656 c	49.105 mno	0 c	0.568 ıijk	0.640 ghı	70.670 a	-2.150 c	7.653 v
12	0 g	8.487 ıij	33.855 fg	56.419 ghı	0 c	0.561 ıijk	0.644 ghı	70.527 abcd	-1.857 a	6.783 y
13	0 g	8.635 ıij	34.467 ef	55.406 hıi	0 c	0.524 ıijk	0.649 ghı	70.533 abcd	-2.530 hı	9.447 ö
14	0.038 ef	7.125 jkl	30.536 ijklmn	61.267 abcd	0 c	0.528 ıijk	0.704 fghı	70.403 cdef	-2.297 de	8.830 t
15	0 g	5.330 oö	29.133 klmno	64.480 a	0 c	0.648 ghıijk	0.657 ghı	69.827 ıijkl	-3.230 s	11.997 h
16	0 g	16.219 de	24.608 rs	57.652 efgh	0 c	0.941 defghı	0.730 fghı	70.430 abcdef	-1.903 a	7.597 v
17	0.040 e	13.417 fg	26.297 öpr	59.149 bcdefg	0 c	0.638 hıijk	0.610 hı	70.660 ab	-2.610 j	9.703 o
18	0.053 e	7.416 jklm	37.286 e	54.279 ıij	0 c	0.464 jk	0.632 ghı	69.887 hıijk	-2.407 fg	9.213 p
19	0 g	8.232 ıijk	31.736 ghıij	58.568 cdefg	0 c	0.548 ıijk	0.698 fghı	69.597 l	-3.010 ö	11.090 i
20	0 g	12.344 gh	30.751 ıijklmn	55.303 hıi	0 c	1.084 bcdefg	0.727 fghı	70.420 bcdef	-2.563 ıi	9.780 no
21	0 g	14.707 ef	28.657 mnoö	54.583 ıij	0 c	1.112 bcdef	0.732 fghı	70.163 efg	-2.720 l	10.037 m
22	0 g	16.465 d	33.662 fgh	47.653 noöp	0 c	1.265 bcd	0.740 efghı	69.773 ijkl	-3.643 ü	14.553 d
23	0 g	17.237 d	30.753 ıijklmn	50.470 klm	0 c	1.027 cdefgh	0.752 efghı	70.440 abcdef	-2.730 l	10.093 m
24	0 g	14.232 ef	31.222 hıijkl	52.481 jk	0 c	1.426 abc	0.682 fghı	70.513 abcd	-3.133 r	12.210 g
25	0 g	21.377 bc	23.522 s	52.640 ijk	0 c	1.345 bcd	0.810 defgh	69.660 kl	-2.013 b	8.257 ü
26	4.055 a	11.530 h	33.265 fghı	49.721 lmn	0 c	0.733 fghıijk	0.669 fghı	70.297 cdef	-2.843 m	10.573 l
27	0 g	22.614 b	28.394 noö	46.557 oöp	0 c	1.210 bcde	0.874 def	70.100 efgh	-2.940 o	10.737 k
28	0.054 e	5.678 mnoö	46.585 c	46.406 öp	0 c	0.549 ıijk	0.664 ghı	70.567 abc	-2.753 l	9.740 no

29	0 g	14.781 ef	33.259 fghı	50.478 klm	0 c	0.899 defghıi	0.709 fghı	70.550 abc	-2.347 ef	9.053 rs
30	0 g	6.622 klmn	58.456 b	26.634 r	5.251 b	2.058 a	1.169 bc	69.917 hıij	-4.233 y	16.060 c
31	0 g	14.427 ef	25.669 prs	58.113 defgh	0 c	1.110 bcdef	0.659 ghı	70.523 abcd	-2.413 g	9.767 no
32	0 g	22.327 b	28.820 lmno	46.459 oöp	0 c	1.123 bcdef	0.744 efghı	69.177 m	-3.403 t	12.967 e
33	0 g	21.618 bc	30.509 ijklmno	45.538 p	0 c	1.104 bcdef	0.816 defg	70.630 abc	-2.263 d	8.850 şt
34	0 g	20.440 c	23.464 s	49.369 mn	5.295 b	1.115 bcdef	0.339 l	70.573 abc	-3.333 ş	11.773 ı
35	0 g	11.518 h	28.450 mnoö	53.400 ij	5.400 b	0.734 efghıijk	0.357 k	70.233 defg	-3.470 u	12.530 f
36	0 g	9.199 ı	37.226 e	52.344jkl	0 c	0.578 ıijk	0.564 ıi	70.217 defg	-3.077 p	11.827 ı
Cottonseed oil	0.502 c	28.268 a	16.533s	53.392ij	0 c	0.818 efghıij	0 m	66.903 n	-4.120 v	32.057 b
Canola oil	0 g	5.475 noö	68.251a	17.414s	7.557 a	1.547 ab	0 m	64.933 o	-5.893 z	53.973 a

*Means with different superscript letters differ significantly.

The samples, 1, 2, 3, 8, 9, 10, 11, 14, 15, 18, 28 and 30 were found to be in the range in palmitic acid as mentioned in the regulation (4,0 - 7,6 %) announced by the Ministry. The palmitic acid content was ranged between 8,232 % - 9,200 % for the samples 4, 5, 6, 7, 12, 13, 19 and 36. The samples, 17, 20, 21, 24, 26, 29, 31 and 35's palmitic acid content were detected to be from 11.530 % to 14.781 %. It was surprising to detect the palmitic acid content of the samples 16, 22, 23, 25, 27, 32, 33 and 34 in between 16,219 % and 22,327 %. This classification aroused the suspicion of adulteration of sunflower oil with cottonseed oil for the last group, due to higher amount of palmitic acid.

Oleic acid content of sample 30 was 58,456 % which was found to be higher than other sunflower oil samples. Oleic acid content of samples 10, 11 and 28 were from 40,863 % to 46,585 %. The lower range of oleic acid content was from 23.464 % to 26.297 % for the samples 16, 17, 25, 32 and 34. The range for oleic acid content in sunflower oil, canola oil and cottonseed oil was announced as 14,0 - 71,8 %, 51.0-70.0 % and 14,7 - 21,7 %, respectively. Estimation of adulteration by the aid of data on oleic acid content of sunflower oils looks too hard to evaluate the suspicion of addition of canola and cottonseed oil.

The lowest linoleic acid content of sample 30 was 26,634 %. The linoleic acid content of sunflower oil, canola oil and cottonseed oil in the regulation announced by the Ministry was ranged as 18,7 - 74,0 %, 15,0 - 30,0 % and 46,7 - 58,2 %, respectively. Linoleic acid content of sample 22, 27, 28, 32, 33 and 34 was from 45,538 to 49,369 %. The other sunflower oil samples were detected to have a linoleic acid range in between 48,427 and 64,480 %. In general, the linoleic acid content of sunflower oil and cottonseed oil is similar and it is not possible use the linoleic acid data as estimation parameter on adulteration of sunflower oil by cottonseed oil. But the addition of canola oil in sunflower oil may cause a little decrease in linoleic acid content of sunflower oil.

Linolenic acid may be a good estimation parameter for addition of canola oil in sunflower oil due to apparent increase in percentage. In the announce of the Ministry's regulation, the range for linolenic acid was from 0 to 0,5. The detection of linolenic acid in sample 30, 34 and 35 was from 5,251 to 5,400 % that increases the suspect of canola oil addition in sunflower oil. If linolenic acid content is taken in to account, the possibility of estimation of cotton seed oil addition in sunflower oil is very poor due to low ranges of linolenic acid content in cottonseed oil (0 - 0,4 %). Detection of linolenic acid in sunflower oil arouses the suspicion of adulteration of sunflower oil with canola oil due to a visible

increasement. Linolenic acid was not detected in the sunflower oil samples except for the samples 30, 34 and 35.

Arachidic acid content of sample 30 was found as 2, 058 % and was higher than the other sunflower oil samples. It was the sample that was highly suspected to be adulterated with canola oil by the data on linolenic acid. The arachidic acid data was the second hint to strength this suspicion for the sample 30. The arachidic acid content of samples 4, 6, 20, 21, 22, 23, 24, 25, 27, 31, 32, 33 and 34 was from 1,084 to 1,459 %. These data on arachidic acid are higher than the announcement of the Ministry (0,1 - 0,5 %) for sunflower oil. According to these results, it may be offered to the Ministry to increase the limits of arachidic acid content up to 1,5 % in sunflower oil.

Behenic acid content of all tested samples was in the range that Ministry announced. Behenic acid is not a good parameter for estimation of adulteration of sunflower oil with the addition of canola and cottonseed oil.

DISCUSSION

Raw canola and cottonseed oils are cheaper than sunflower oil. By this study the suspense of adding these cheaper oils in sunflower oil was inspected by the evaluation of the possibility of the usage of fatty acid composition as a verification parameter.

Detection of linolenic acid in sunflower oil may strength the suspense of adding canola oil in suflower oil. Palmitic acid content increases by the addition of cottonseed into sunflower oil. Sample 30 is a special example that may be announced to be the most suspected sunflower oil to be adulterated by the addition of canola oil, individually. Linolenic acid was detected in sample 30 and also the amount of oleic acid was relatively higher enough to strength the possibility of suspicion. The palmitic acid and linolenic acid content of sample 34 and 35 were higher in comparison to other sunflower oil samples those shift the tendency of suspense on addition of both canola and cottonseed oils. Especially *b** value was found to be the highest for the sample 30. b* was also high in samples 34 and 35. Those oils were thought to be most suspected ones among the other samples which could be adulterated.

Detailed statistical modeling studies may be recommended for estimation of cheaper raw oil addition in sunflower oil. Detection of linolenic acid in sunflower oil may be a good indicator for addition of any other oil, especially the addition of canola oil. Palmitic acid may be a parameter for estimation of cottonseed addition but it is not a strong indicator individually. b* value was found to be high in the sample which was the most suspicious to be adulterated with the addition of raw canola oil. *b** value of other suspected samples were also high in comparison to the other sunflower oil samples. Additionally, revision of the arachidic acid range of sunflower oil in the related regulation may be referred to Ministry to increase the upper limit up to 1,5 %.

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EFFECT OF DIFFERENT STORAGE CONDITIONS ON QUALITY PROPERTIES OF RAW AND ROASTED SUNFLOWER KERNELS

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ABSTRACT

Quality of raw sunflower kernels changes due to the biochemical changes throughout the storage period. Thus, quality of sunflower kernels (SK) roasted after different storage periods may have different shelf lives. Relative humidity and temperature are the main factors affecting the quality of raw SK, whereas packaging material (O2 and water vapour barrier) properties and the gas composition in the package are the main factors affecting the quality of roasted sunflower kernels. The purpose of the present study was to explore the influences of storage conditions (room conditions-LOCAL and 10°C, Relative Humidity<%65 - MAM) on the quality of raw SK and to extend our knowledge concerning the changes in oxidative stability of roasted sunflower kernel processed at various storage periods (just after harvest, 8 and 12 months after harvest). Roasted products were packed in packaging material with high oxygen barrier (<0.008 ml/m²/day at 23°C) properties and kept at 10, 20 ve 30°C storage conditions under normal atmospheric conditions and nitrogen gas (>95%). Peroxide value, free fatty acids, contents of hexanal and vitamin E were determined at 2 months intervals during the storage for 12 months. Oxidative quality of the raw SK was similar when stored at cool (10°C, RH<65%) and local conditions (avg. 51 %RH, 19°C). SK roasted at 8th and 12th month storage periods lost quality more rapidly than the kernels roasted just after the harvest. Packaging under nitrogen gas rather than cold storage had the strongest influence in the prevention of oxidative changes of the roasted products.

Key Words : Sunflower kernel, oxidation, rancidity, peroxide value, free fatty acid, hexanal, vitamin E

QUALITY CHARACTERISTICS OF ROASTED SUNFLOWER SEEDS DURING STORAGE

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ABSTRACT

Sunflower seed being a part in both oil and dried nut industry is a highly nutritious oil seed. The oil and unsaturated fatty acids content plays an important role in determining the shelf life of seeds depending on lipid oxidation while increasing the nutritional value of the seeds. The oil seeds, like sunflower seed, which have high unsaturated fatty acid content, are exposed to oxidation during the long time storage that cause off-flavour, taste and rancidity. This may result in reduced overall sensory score when consumed. The packaging material properties (oxygen and water vapour permeability) have important effects on the shelf life of roasted dried nut products. The main objective of this study is to investigate the quality changes of sunflower seed in different packaging conditions and to optimize storage conditions for longer shelf life. In this study, the sunflower seeds obtained from different planting areas (Ankara, Kayseri, Bursa-İnegöl) were first roasted and then packaged under atmospheric and nitrogen gas conditions, and stored at 20°C for estimation of the shelf life. Peroxide value, free fatty acids, hexanal content, Vitamin E content and sensory quality properties were monitored during the shelf life study. As a result of this study; bio-chemical and sensory qualities of the stored products decreased within 2 months of storage period. It was observed that the product which is obtained from Bursa-İnegöl planting area packaged under nitrogen has the best chemical and sensory quality properties.

Key Words : Sunflower seed, oxidation, peroxide value, hexanal, Vitamin E, sensory

ACCEPTABILITY OF CHAPATI MADE WITH SUPPLEMENTATION OF SUNFLOWER (*HELIANTHUS ANNUS* L.) SEED MEAL

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ABSTRACT

The nutritional value of lab processed sunflower seed meal prepared from different sunflower seed cultivares i.e HSFM-848 and Morden as well as commercially processed cake (CPC) of sunflower seeds.*Chapati* was standardized in the lab by addition of sunflower seed meal and protein isolates (obtained from CPC) at 10,20 and 30% level. Nutritional evaluation revealed that lab processed seed meal of HSFM-848, Morden and CPC contained crude protein 42.51,51.44 and32.66%, fat 1.48, 0.86 and 0.55% crude fibre 4.16,2.48 and 14.56%, calcium 170.00,224.00 and 192.33mg/100g and iron 4.28, 25.12 and 22.13 mg/100g, respectively. Lab processed meals had significantly lower amount of polyphenols and higher amount of saponins as compared to the value of CPC. *in vitro* protein digestibility of lab processed seed meal as well as CPC was found to be improved after processing. *Chapatis* were found to be organoleptically acceptable. All the developed *chapatis* were rated in the range of like moderately to like very much category on Nine-Point Hedonic scale. Incorporation of sunflower seed meal and protein isolates at 10% level with wheat flour was the desirable level without altering the organoleptic traits and can be used for preparation of other traditional products like halwa suhali,cake & biscuits. These sunflower seed meal supplemented products if added in children diet can help in over coming protein energy malnutrition among infants & children in india.

Key Words : supplements, nutritional value, sunflower seed meal, acceptability, chapatti

SOME ANTINUTRIENTS AND IN VITRO PROTEIN DIGESTIBILITY OF HOME PROCESSED SUNFLOWER SEED MEAL

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ABSTRACT

Lab processed seed meals obtained from HSFM-848 was prepared by decorticating the seeds manually followed by grinding and extracting the oil with Hexane. Commercially Processed Meal contained average polyphenol content of 1675.00, 2001.66 and 1945.00 mg per 100g, saponin content of 1935.35, 1420.75 and 1112.25 mg per 100g respectively. Polyphenol content of CPC was significantly higher than those of Lab processed seed meals, whereas saponin content of CPC was significantly lower than those of lab processed seed meals Lab processed seed meals prepared from HS-1 and Morden cultivars and commercially processed cake contained on the average crude protein content (41.75, 50.68 and 31.75%), fat (1.45, 0.95 and 0.45%), crude fibre (3.75, 2.12 and 14.85%), respectively. But the polyphenol content of commercially processed cake (1936.00 mg/100g) was found to be significantly higher than those of both the lab processed seed meals. Saponin content of lab processed seed meal prepared from HSFM-848 variety (1922.68 mg/100 g) was significantly higher than that of Morden variety whereas the saponin content of commercially processed cake (1112.65 mg/100g) was found to be significantly lower than that of both the lab processed seed meals.It may be concluded from the study that the seed meal obtained from sunflower seeds after laboratory processing is nutritionally superior, in the preparation of various traditional food products. These food products if added in the diet will improve the nutritional quality of home diet .Processing has a significant effect on lowering antinutrients present in sunflower seeds which results in increase of *in vitro* digestibility of proteins and availability of minerals from sunflower seed meal.

Key Words : In vitro protein digestibility, home processed, sunflower seed, saponins, polyphenol

CONTENT AND OIL PRODUCTIVITY IN SUNFLOWER GENOTYPES PRODUCED IN CAMPO NOVO DO PARECIS – MT, BRAZIL

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ABSTRACT

This study aimed to evaluate genotypes of sunflower seeded second harvest in the year 2014 in Campus Campo Novo do Parecis, in the experimental field of the Instituto Federal de Educação Ciência e Tecnologia de Mato Grosso. The experimental design was a randomized block design with treatments 16 (16 genotypes) and four replications. The experimental plots consisted of four rows 6.5 m long with row spacing of 0.45 m, containing area of 11.7 m², totaling an area of 748 m². The population of 45000 plants per hectare is used. Data were subjected to analysis of variance and the Scott - Knott test at 5 % probability. The genotypes that stood out in relation to achenes productivity were the MG 360, AGUARÁ 06, MG 305, AGUARÁ 04, CF 101, SYN 045, GNZ NEON, HELIO 251 and SYN 3950HO. For the achenes oil content and productivity, the MG 360 genotype was the highest value and stands in relation to other genotypes.

Keywords: spectroscopy, *Helianthus annuus* L., lipids, oilseeds, achenes productivity.

INTRODUCTION

Among oilseeds grown in the world, the sunflower stands out among the main, both in production and in planted area. Sunflower (*Helianthus annuus* L.) is an annual cycle plant and its rapid growth characteristics, resistance to drought, cold and heat, more than most species of economic cultivation in Brazil and can be used for various purposes (Leite et al., 2005) as high quality oil extraction for human consumption or as raw material for biodiesel production, among others.

In general, sunflower seed it has about 45 to 65% oil in its composition (Grunvald et al., 2014A). Sunflower oil essentially consists of triglycerides (98 to 99%). It has a high content of unsaturated fatty acids (about 83%) and Vitamin E (alpha-tocopherol), but a reduced content of linolenic acid (≤ 0.2%). Sunflower oil is essentially rich in essential fatty acid (EFA) linoleic acid, about 60% that helps in reducing serum cholesterol and LDL. Thus contributing to the prevention of arteriosclerosis and cardiovascular problems (Turatti et al., 2002).

Changes in oleic are the result not only of the genotype, but also of climatic differences during their cultivation. Thus, among the various technologies developed for sunflower production, the appropriate choice of the genotype that has high yield and / or oil is important to ensure the success of the culture as a component of the production system (Porto et al., 2007).

In the region of Campo Novo do Parecis, sunflower is grown second summer harvest from February/March, due to the occurrence of rainfall conditions and temperatures suitable for its cultivation (Castro and Farias, 2005). However, despite being the main growing region in the country, little information is available on the agronomic characteristics of genotypes as content and productivity of oil, to facilitate the cultivation practices, reducing risk and increasing profitability.

MATERIAL AND METHODS

The work was carried out at the experimental fields and facilities of the Instituto Federal de Educação Ciência e Tecnologia de Mato Grosso - Campo Novo do Parecis in second-crop system in succession to soybeans in the agricultural year 2013/2014. The soil, according to the American System of Soil Classification (USDA, 1960) is the Typic Tropudox. The initial characterization of fertility, for the first layer of 0-0.20 m, presented the following values: pH ($CaCl_2$) = 5,7; MO = 26 g dm^{-3}; P (resina) = 5,9 mg dm^{-3}; K, Ca, Mg e H+Al = 1,5; 32; 11 e 40 $mmol_c$ dm^{-3}, respectively; with V = 54,8%.

Average temperatures occurred during the experimental period were: 30.3; 23.2 and 18.9 °C for maximum temperature, medium and minimum, respectively, and 570 mm rainfall, meeting the water demands required by sunflower between 500 and 700 mm distributed along its growing cycle (Castro and Farias, 2005).

The experimental design was a randomized complete block design with 16 treatments (genotypes) and four replications, as follows: ADV 5504, AGUARÁ 04, AGUARÁ 06, BRS 323, BRS G42, CF 101, GNZ NEON, HELIO 250, HELIO 251, HLA 2012, M734, MG 305, MG 360, PARAISO 20, SYN 045 and SYN 3950HO. The experimental plots consisted of 4 rows with 6.5 m long, with row spacing of 0.45 m, containing area of 11.7 m² (1.8 x 6.5 m). Only the two 5 meters central rows of each genotype were considered for data collection. The plotted area comprises 4.5 m².

The plot of the rows, was done on March 7, 2014, and the previous application of fertilizers was carried out with the aid of a sowing machine and was distributed at a depth of 0.10 m, 45 kg ha^{-1} Potassium Chloride + 267 kg ha^{-1} NPK 10-30-20, totalizing: 26.7 kg ha^{-1} N; 80 kg ha^{-1} P2O5; 80 kg ha^{-1} K_2O, according to the results of soil analysis and recommendation (EMBRAPA, 2004). Further, beside the row fertilization at 0.04 m deep, three seeds were placed in each hole, each 0.495 m, by manual planter.

The desiccation and the application of boron was performed on March 07, using trawl trailed sprayer with an application volume of 150 L ha^{-1} using glyphosate (648 g a.i. L^{-1}) at a dosage of 2 L ha^{-1} + Prometryn dosage 2 L ha^{-1} + mineral oil (0.5 L ha^{-1}) + boric acid dosage of 3 kg ha^{-1} (600 g ha^{-1} Boron).

Thinning was done 10 days after emergence (DAE) with a scissor, leaving only one plant per hole, reaching a population of 45,000 plants ha^{-1}.

The following coverage fertilizations were made: 1) 32 DAE with a dosage of 50 kg ha^{-1} N (urea); 2) foliar application of boron, with knapsack sprayer at 35 DAE using a dosage of 3 kg ha^{-1} (600 g ha^{-1} Boron), and 43 DAE with a dosage of 11 kg ha^{-1} (1.1 kg ha^{-1} of Boron). The source of Boron used was boric acid 150 L ha^{-1} according to the requirement of sunflower of 2 kg ha-1 B, Control of weed, pests and diseases have been carried out according to the recommendations of EMBRAPA (2004).

To avoid birds attacks, the plotted sections of the central rows were protected (stage R6) by using polypropylene based bags (30 x 30 cm) and fixed with clips.

The following agronomic characteristics were evaluated: productivity achenes (**PR**; kg ha-1), determined based on two central lines 5 meters, which is corrected for moisture condition of 11% (wet basis) obtained by reading the humidity value of the achenes; oil content (**OC**; %), predicted by near infrared spectroscopy (NIR) according to the methodology described by Grunvald et al. (2014b); and oil yield (**OY**, kg ha-1), calculated by multiplying the achenes oil content (%) and productivity achenes (kg ha-1) / 100.

The harvest of the capitulums was performed manually in the two of 5 meter central rows in R_9 with pruning shears aid. Later the capitulum inflorescence were the natural dryied, cleaned and weighed. The results were submitted to analysis of variance followed by the average test Scott-Knott, both 5% probability, with the statistical program SISVAR (Ferreira, 2011).

RESULTS AND DISCUSSION

All variables showed significant differences ($p < 0.05$) in the analysis of variance (Table 1). The data from the achenes productivity variables, oil content and oil yield are shown in Table 2. For the achenes productivity, genotypes were stood SYN 3950HO (2205.5 kg ha^{-1}) and HELIO 251 (2204.1 kg ha^{-1}), but not statistically different genotypes GNZ NEON, SYN 045, CF 101, AGUARÁ 04, MG 305, AGUARÁ 06 and MG 360, which had average productivity ranging from 1836.8 e 2132.5 kg ha^{-1}. However, it appears that even the lowest yields were found in genotype HLA 2012 e BRS G42, with average 40% lower than those observed in the most productive genotypes.

Table 1. ABSTRACT of the analysis of variance for the sunflower productivitiy parameters (Campo Novo do Parecis, MT, 2014).

Parameters[1]	F[2]	CV (%)[3]	GA[4]
PR (kg ha^{-1})	6.4*	12.4	1846.9
OC (%)	27744.6*	0.1	43.2
OY (kg ha^{-1})	6.8*	12.4	796.5

[1] PR = achenes productivity, OC = oil content, OY = oil yield; 2 * significant at 5%; 3 CV = Coefficient of variation; 4 GA = General average.

Values higher than this study were found by Backes et al. (2008) for HELIO 250 genotypes (1849.0 kg ha^{-1}), M734 (2052.0 kg ha^{-1}), AGUARÁ 04 (2252.0 kg ha^{-1}) and below to HELIO 251 (1882.0 kg ha^{-1}) in second-crop cultivation in northern Santa Catarina. Additionally, Vogt et al. (2010), in sunflower crop sown in November in northern Santa Catarina, reported higher yields for genotypes AGUARÁ 04 (1916.0 kg ha^{-1}) e M734 (1962.0 kg ha^{-1}) and means inferior to HELIO 250 (1450.0 kg ha^{-1}). Already Capone et al. (2012) evaluated the performance of cultivars in southern Tocantins state reported productivities 2834.1 e 2997.6 kg ha^{-1} para os genótipos HELIO 250 e HELIO 251, respectively. Poletine et al. (2013) reported an assay developed in the northwestern region of the state of Paraná, for genotypes BRS G42, SYN 3950HO, M734 and MG 305, with productivities 715.5 kg ha^{-1}, 1215.0 kg ha^{-1}, 1225.0 kg ha^{-1} e 1592.0 kg ha^{-1}, respectively. These variations in productivity reveal the importance of evaluation of genotypes in different producing regions to verify the feasibility of its use.

Analyzing the oil content of genotypes, the MG 360 genotype had the highest oil content, 47.8% (Table 2), differing from the other investigated genotypes.

Table 2. Mean values for productivity achenes (PR), oil content (OC) and oil yield (OY) from different sunflower genotypes.

Genotypes	PR (kg ha^{-1})	OC (%)	OY (kg ha^{-1})
ADV 5504	1446.9 c	47.1 b	681.5 b
AGUARÁ 04	2084.1 a	45.9 d	956.6 a
AGUARÁ 06	1859.5 a	41.6 n	773.7 b
BRS 323	1782.0 b	42.1 l	750.2 b
BRS G42	1425.9 c	42.0 m	598.9 b
CF 101	2104.4 a	45.1 f	949.1 a
GNZ NEON	2132.5 a	37.8 p	806.1 a
HELIO 250	1694.7 b	43.5 h	737.2 b
HELIO 251	2204.1 a	39.1 o	861.8 a
HLA 2012	1313.0 c	46.7 c	613.2 b
M734	1673.7 b	37.6 q	629.3 b
MG 305	1993.8 a	43.3 i	863.3 a
MG 360	1836.8 a	47.8 a	878.0 a
PARAISO 20	1685.3 b	43.2 j	728.5 b
SYN 045	2108.5 a	43.6 g	919.3 a
SYN 3950HO	2205.5 a	45.2 e	996.9 a

Different letters differ by Scott-Knott test at 5% probability.

However, the ADV 5504 genotypes (47.1%) and HLA 2012 (46.7%) also showed considerable oil content. In contrast, the M734 genotype was presented the lower oil content, with the representative average 37.6%. Some industries have been remunerating the sunflower producers from the oil content contained in achenes and no longer by simple mass achenes, since not always the genotype with the highest productivity of achenes per area results in greater productivity of oil in the same area , and the oil product of greater interest at the end of the manufacturing process and currently the main commercial sunflower crop product.

Watching the oil yield data, the averages of the genotypes SYN 3950HO, AGUARA 04 CF 101, SYN 045, MG 360, MG 305, HELIO 251 and GNZ NEON were the ones that showed the highest values (Table 2), getting between 806.1 (GNZ NEON) and 996.9 kg ha^{-1} (SYN 3950HO), but all belonging to the same statistical group. Thomas et al. (2012), testing different planting dates mentioned lower oil yield for AGUARA 04 genotypes, with 928.0 kg ha^{-1}, and HELIO 250, with 717.0 kg ha^{-1}. For the M734 genotype, the value was 864.0 kg ha^{-1}.

CONCLUSIONS

For achene productivity variable stood out the AGUARA 04 and 06 genotypes, CF 101, GNZ NEON, HELIO 251, MG 305 and 360 and 045 and SYN 3950HO, whose values were ranging between 1836.8 and 2205.5 kg ha^{-1}. However, for the oil content of the MG 360 was the one with the highest percentage, especially also in the group of genotypes with the highest oil productivity values, confirming its high potential for use in production systems Brazilian savannah.

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DETERMINATION OF FATTY ACID COMPOSITION FOR FRYING SUNFLOWER OIL USING GAS CHROMATOGRAPHY

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ABSTRACT

Frying of sunflower oil has been carried out for 7 running days at 175°C±2 in this study. The aim of this study is to determine fatty acid composition of sunflower oil under real domestic frying conditions. In the frying processes, potato has chosen for food and the processes have continued during seven days. The composition, trans fatty acid (TFA) amount and average molecular weight of sunflower oil have been determined by gas chromatography (GC) technique. This work focuses on finding changes in free fatty acid after repeated batch potato frying. Unsaturated fatty acid (UFA) contents of sunflower oil have been decreased and saturated fatty acids (SFA) have also been increased during frying process. When the frying times run on, the analysis of oil samples have showed that trans fatty acids such as elaidic and linoelaidic acids occur quite slowly. At the end of the repeated frying series, the elaidic acid (C18:1 trans) has been determined in oils for sunflower 1.5%. And also linoelaidic acid (C18:2 trans) has been detected 0.3%. At the end of the seventh day of frying, the relative contribution of oleic acid has been decreased.

Key words: Sunflower, frying, fatty acid composition

INTRODUCTION

Today, frying is one of the most popular methods for the preparation of food stuff, because the method is fast and relatively cheap and results in yellow brown products with a typical taste and smell, preferred by the consumer. The oil plays a critical role as a heat transfer and impregnation medium, and it is the crucial component of the frying process. For the quality of products being fried the quality of the frying medium is very important, because during frying the food takes up the oil becoming a significant part of the product. (Taha et al., 2014). Many factors affect the deterioration of a frying oil, such as the presence of unsaturated fatty acids, the oil temperature, oxygen absorption, the presence of metals, and the type of food (Arroyo et al. 1992). During frying, oil or fat is subjected to high temperatures in the presence of air and water from the food, thus producing a wide range of compounds resulting from thermal, oxidative, and hydrolytic reactions (Chatzilazarou et al. 2006, Dobarganes et al. 2013). As a result of the deterioration, the oil sustains some physical changes: the colour darkens, the viscosity increases, and smoke appears (Paul and Mittal, 1997).

The fatty acid composition of the frying oil is an important factor affecting fried food flavor and its stability; therefore, it should be low level of polyunsaturated fatty acid such as linoleic or linolenic acids and high level of oleic acid with moderate amounts of saturated fatty acid (Kiatsrichart et al., 2003, Mehta and Swinburn, 2001). Partial hydrogenation decreases polyunsaturated fatty acid but increases saturated fatty acid and trans-fatty acid to

produce more stable frying oil. However, trans-fatty acids (TFA) adversely effects on cardiaovascular health (Rehab and Anany 2012). One approach to increasing the stability of unsaturated oil is partial hydrogenation (Li et al., 2008; Bysted et al., 2009), but hydrogenation also results in the formation of SFA and trans fatty acids. Trans isomers of fatty acids have been reported to increase the ratio of low-density-lipoprotein (LDL) to high-density-lipoprotein cholesterol (HDL) in the plasma and increase the risk of coronary heart disease (CHD), and play a part in atherosclerosis development (Willett et al., 1993; Dalainas and Loannou, 2008). Low levels of trans fatty acids and saturated fatty acids that are basis of nutritional and diet physiological aspects also play important roles in selecting a frying oil. Since the fatty acid composition alone is not enough to explain the stability of oils, a variety of minor components, such as tocopherols, polyphenols, phospholipids, caretonoids and certain sterols are also beneficial to oil stability during frying (İnanç and Maskan 2012).

Oil and fats are one of the important components of human diet and ingredients of food industry. Oils and fats are preferred as carriers of fat soluble vitamins (A, D, E and K) and source of essential fatty acids and energy (Öğütcü et al., 2015). Vegetable oils are recognized as important compounds of our life. Sunflower is between the five biggest oilseeds in world production (Anwar et al., 2008). Sunflower oil contains a wide range of unsaturated fatty acids and is rich in essential fatty acids. Sunflower oil is considered nutritious due to high content of polyunsaturated fatty acids (PUFA), mainly linoleic acid (18:2). However, due to high PUFA, it is more susceptible to oxidative degradation leading to rancidity, off-flavors, and discoloration (Gordon 1991). And also sunflower oil is characterized by high content of tocopherols (up to 935 ppm) higher than those of other oils such as soybean and peanut. It is considered an oil of high stability due to its high content in natural antioxidants (Bramley et al., 2000; Shahidi, 2005). The nutritional aspects of edible oils associated with the presence of minor and major components play an important role in preventing diseases and improving health. It is important to formulate vegetable oil blends with special composition in order to enhance their stability and nutritional value (Frankel et al., 1994; Shiela et al., 2004).

The objective of the present study was to obtain the fatty acids combination of refined sunflower oil under normal frying conditions. Frying processes were done with potato repeating seven days.

MATERIALS AND METHODS

Frying Process

At the beginning of frying, the fryers have been stuffed with 2 L of fresh oil samples, and then oils have been heated to 175 ± 2 °C. The frying temperature has been controlled using a probe joined to the thermometer. An electrical domestic deep-fat fryer has been used for frying experiments. Prior to frying, potato slices have been dried on both sides on filter paper to remove any excess water. The frying process started 30 minutes after the temperature reached at 175 ± 2 °C. The frying time has been 6 minutes for potato slices. One frying has been done per day for seven consecutive days. All physical and chemical analyses of oils have been performed immediately after the frying. During frying process, fresh oil has not been added to frying pans.

Determination of Fatty Acids Composition

Gas chromatography has been used for the qualitative and quantitative determinations of the fatty acids reported in relative area percentages. Fatty acids have been methylated prior to analysis by gas chromatography. Analysis have been performed on Agilent 9C 6890N gas chromatograph (CA, USA) equipped with a DB-23 capillary column (60 m, 0.32 mm, 0.25μm

film thickness) and a flame ionization detector. The oven temperature has been arranged from 160°C to 185°C at a rate of 7 minutes, later programmed from 195°C to 220°C for 3 minutes, finally kept 20 minutes at the last temperature. The injector and detector temperatures have been 230°C and 255°C, respectively. Nitrogen has been used as carrier gas at a flow rate of 1.0 ml/min. FAME has been identified by comparing their retention time with known commercial standard mixtures.

RESULTS AND DISCUSSION

The fatty acid compositions of sunflower oils are shown in Table 1. Composition of fatty acid in sunflower oil contained palmitic acid (7.1 %), stearic acid (4.3 %), oleic acid (19.0 %), linoleic acid (67.5 %) and linolenic acid (0.8 %). These results belong to before starting fryings. Linoleic acid (C18:2) is determinated the most abundant unsaturated fatty acid in the sunflower oil. Linolenic acid (18:3) is highly sensitive to oxidation because it contains three double bonds, while oleic acid (18:1) is less reactive as it contains only one double bond. At the end of the frying processes, composition of fatty acid in sunflower oil contained palmitic acid (11.4 %), stearic acid (4.9 %), oleic acid (9.1 %), linoleic acid (47.9 %) and linolenic acid (0.0 %). It is observed that there is a decrease in polyunsaturated fatty acids and resulting increase in the saturated acids content. When the frying times run on, the analysis of oil samples have showed that trans fatty acids such as elaidic and linoelaidic acids occur quite slowly. The elaidic acid ($C18:1_{trans}$) has been determined in oils for sunflower %1.5. And also linoelaidic acid ($C18:2_{trans}$) has been detected 0.3%. At the end of the seventh day of frying, the relative contribution of oleic acid has been decreased for sunflower oils.

Poor frying stability in sunflower oil comes primarily from the high level of linoleic acid. Therefore, sunflower oil must also be hydrogenated to reduce its linoleic acid content to 35% or lower for industrial frying. On the other hand, fatty acid compositions do not fully explain frying stability of oils. For understanding of the frying stability of oil, there are so many parameters. Stability of oil indicates that the oil must be low in free fatty acids, peroxide value, conjugated dienes, anisidine value, monoacylglycerols, diacylglycerols, and trace impurities, such as iron, phosphorus, calcium, and magnesium. All of these quality parameters have specific significance in influencing the performance of the frying oil.

Table 1 Changes in fatty acid composition (%) during frying processes.

Fatty Acids	Fresh oil	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day
$C_{14:0}$	0.1982	0.2472	0.4000	0.5772	0.7579	0.9120	1.0700	1.2035
$C_{15:0}$	-	-	-	0.1859	0.3580	0.4727	0.5674	0.6498
$C_{15:1\ cis}$	0.2683	0.2037	0.1229	0.0680	0.0391	0.0253	0.0157	0.0102
$C_{16:0}$	7.0560	7.4007	8.0065	8.4157	9.2316	10.0710	10.8954	11.3895
$C_{16:1\ cis}$	0.1731	0.1710	0.1698	0.1687	0.1654	0.1619	0.1559	0.1463
$C_{16:1\ trans}$	-	-	-	-	-	-	-	-
$C_{17:0}$	-	-	-	-	-	-	-	-
$C_{17:1\ cis}$	-	-	-	-	-	-	-	-
$C_{18:0}$	4.3061	4.4458	4.5834	4.7435	4.8403	4.9146	4.9414	4.9502
$C_{18:1\ cis}$	18.9617	18.1325	17.2031	16.3104	15.1967	13.5689	11.2314	9.1256
$C_{18:1.\ trans}$	-	-	0.139	0.4793	0.7193	0.9486	1.2546	1.4876
$C_{18:2\ cis}$	67.5091	63.1032	59.9364	56.0213	53.4558	51.0132	49.0135	47.9356
$C_{18:2\ trans}$	-	0.0601	0.1147	0.1625	0.2053	0.2421	0.2748	0.3059
$C_{18:3\ cis}$	0.7778	0.5364	0.4915	0.3221	0.2287	0.2032	0.0913	-
$C_{18:3\ trans}$	-	-	-	-	-	-	-	-
$C_{20:0}$	0.2939	0.3192	0.3605	0.3989	0.4408	0.4854	0.5073	0.5231
$C_{20:1\ cis}$	0.1552	0.1187	0.0983	0.0812	0.0706	0.0567	0.0364	0.0286
$C_{20:1\ trans}$	-	-	-	-	-	-	-	-
$C_{20:2}$	-	0.0102	0.0243	0.0411	0.0618	0.0825	0.1026	0.1168
$C_{20:3}$	-	-	0.0306	0.0052	-	-	-	-
$C_{20:5}$	0.062	0.0245	0.0056	-	-	-	-	-
$C_{22:0}$	0.6325	0.6726	0.7094	0.7532	0.7831	0.8029	0.8203	0.8316
$C_{22:1}$	0.0153	0.0102	0.0044	-	-	-	-	-
$C_{23:0}$	0.0447	0.0635	0.0976	0.1368	0.1732	0.2123	0.2419	0.2604
$C_{24:0}$	-	0.1201	0.2032	0.2713	0.3404	0.3941	0.4402	0.4657
$C_{24:1}$	-	0.0223	0.0445	0.0614	0.0727	0.0802	0.0889	0.0901

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DETECTION OF REFINED MAIZE AND CANOLA OIL IN COLD-PRESSED SUNFLOWER OIL BY USING RAMAN SPECTROSCOPY

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ABSTRACT

The use of Raman spectroscopy for determination of food adulterations and fraudulent activities has received increased attention due to its simplicity and short response time in last decade. In the meaning of oil analysis, Raman spectroscopic methods positively differ from other analytic techniques such as chromatographic methods which include complex and time consuming sample preparation steps and involve toxic chemicals. Likewise most of the food products seed oils are attractive targets for malpractices. In this paper, the use of Raman spectroscopy for determination of canola and maize oil addition in cold-pressed sunflower oil is presented. The Raman spectra of the oil mixtures containing different amounts of abovementioned oils were collected in the range of 200-2000 cm-1 at a resolution of 5 cm-1. The strongest band at 1645 cm-1 belongs to C=C stretch was same for all three oils but its intensity increased according to the increase in canola oil ratio in the oil mixture. It may due to the highest monounsaturated fatty acid value of canola oil. Sunflower oil and maize oil have closer fatty acid composition compared with canola oil which results to very similar Raman spectra for them. However, some bioactive substances exist in cold-pressed oil affect the Raman signals and the findings showed that refined maize oil addition could be detected using Raman spectroscopy. As a conclusion, in this study, the detection of canola oil in cold-pressed sunflower oil seems possible using Raman spectroscopy at a level of 10 %.

Keywords : maize oil, canola oil, sunflower oil, Raman spectroscopy

DETERMINATION OF REFINED SUNFLOWER OIL IN COLD-PRESSED SUNFLOWER OIL USING RAMAN SPECTROSCOPY

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ABSTRACT

Seed oils are generally subjected to refining process to remove substances which would further catalyse oxidation or other undesirable reactions in commercialized product. On the other hand, cold-pressed oils obtained by mechanical processes without any chemical or heat treatment have been gaining more attendance according to the desire for minimally processed foods. However, cold-pressed oils are attractive targets for fraudulent activities due to their high price compared to refined oils. In this study, existence of refined sunflower oil in cold-pressed sunflower oil was investigated using Raman spectroscopy. Oil mixtures composed of 0-100, 5-95, 10-90, 20-80, 40-60, 60-40, 80-20 and 100-0 % (cold-pressed oil-refined oil) were prepared and analysed with Raman spectrophotometer. The spectra were collected between 200 and 2000 cm-1. The most intense band for both cold-pressed and refined sunflower oils was situated around 1646-1652 cm-1 and 1428-1432 followed this strong band. While the bands in the region of 1400 to 1500 cm-1 have been assigned to =CH2 scissoring, the band situated around 1650 belongs to C=C stretch. There was no missing or new-born band according to the mixture ratios but it was clearly seen that the intensity of the bands were significantly different for every sample. Raman spectroscopy was shown by this study as it is useful for differentiation of refined and cold-pressed sunflower oils. However, there is a need for extended studies to verify present method using more oil samples.

Keywords : Cold-pressed oil, sunflower oil, Raman spectroscopy

MONITORING THE CHANGES IN COLD-PRESSED SUNFLOWER OIL DURING HEATING BY RAMAN SPECTROSCOPY

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ABSTRACT

In the present study, the effect of continuous heating process on Raman spectrum of cold-pressed sunflower oil was monitored. Raman spectra were obtained using DXR Raman Microscopy system with a 532 nm laser source and CCD at 0 °C. Spectra were collected in the range of 200-2000 cm-1 at a resolution of 5 cm-1. Short-term heating process was applied for 30 m on direct heating system and the highest oil temperature was 173 °C measured by oil thermometer. Oil samples were collected in every 5 m through the heating. Continuous heating and especially frying process may result in peroxides degradation and polymerization reactions which are strongly related to the unsaturated fatty acid and other bioactive compounds exist in the oil. Our results showed that the main Raman bands were observed in 860, 960, 1067, 1144, 1255, 1290, 1428, 1509, 1651 and 1737 cm-1. The spectra of the samples collected at different times seemed to be similar. However, there were intensity differences in the same band intensity. Additionally there was a missing band at 1144 cm-1 (-C – O stretching) after 10 m heat treatment. The strongest band positioned at 1651 cm-1 belongs to C=C stretch which tends to loose intensity through the 30 m heating process may be explained due to the high unsaturated fatty acid exist in cold-pressed sunflower oil. As a result, our findings showed that Raman spectroscopy could be used to follow the heat-induced changes in cold-pressed sunflower oil.

Keywords : Sunflower oil, cold-pressed, Raman spectroscopy, heating

APPLICATION OF ARTIFICIAL NEURAL NETWORK ON PREDICTION OF MOISTURE CONTENT OF THE DEEP-FAT FRYING OF BEEF MEATBALLS IN SUNFLOWER OIL

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ABSTRACT:

In this study, it's aimed to predict moisture content of meatball samples fried in sunflower oil. A dataset of temperature, duration of sunflower oil and pH, *L**, *a**, *b**, Chrome, Hue, radius, height of meatballs were processed to develop a three layered artificial neural network. 85% of the objects were used as the training set and 15% as the test set in the application of artificial neural network model. The final developed model presented higher performance for the artificial neural-network than statistical regression model. Artificial neural network is shown to be a powerful and suitable tool for the prediction of moisture content of meatballs fried in sunflower oil.

Keywords: Sunflower oil, deep-fat frying, meatball, artificial neural network, moisture content.

DEEP FRYING QUALITY OF HIGH-OLEIC SUNFLOWER OIL

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ABSTRACT

Sunflower has high content and high quality oil is an important oil plant in all over the World. Sunflower oil has some differences for storage, consumption and industrial quality depending on fatty acid composition. In Turkey, linoleic type sunflower is usually cultivated and also used for industrial purposes. High-linoleic (omega 6) acid content increases to the nutritional quality for sunflower oil. However, this adversely affects to the industrial quality of high-linoleic sunflower because of high oxidative stability. Besides, high-oleic sunflower oil allows higher industrial quality and wide range of uses. Therefore, recent studies deal with breeding the high-oleic sunflower varieties and give to the industry. Frying is a major industrial uses for plant oils. It has critical importance from higher temperature than direct consumption. While polyunsaturated fatty acids (PUFA) are desirable in salads, PUFA is not wanted in frying oils. Therefore, all of the frying oils are originated vegetable have reduced the amount of PUFA through hydrogenation and/or interesterification after refining and hence frying oils have enhanced oxidative stability. Especially high oleic sunflower oil obtained by plant breeding programs, all vegetable oils have reduced content of PUFA is suitable source for frying. This is so important for our country has a very crucial position in sunflower production. Frying is the most preferred method for food cooking and preparing for last 50 years. Deep frying process has 20-200 mm oil height, 5-10 minutes processing time and fried oil is reusable. Firstly, oil is preheated to 150-180 °C for frying process. When the food contact to the oil, surface temperature reaches to the oil temperature rapidly. However, the inner part of food remains between 80-100 °C. Degradation products with hundreds of dissimilar structures occurred with different reactions via varied mechanisms and under varied temperatures. However, all of the degradation products are polar character and deep frying process is often used in fast food restaurants. These oils are subject to chemical and physical changes after 10-12 hours frying. Fried food consumption frequently and continuously increases the risk of cancer and cardiovascular and gastrointestinal diseases. The properties of the oil used in frying process are the biggest factor in the emergence of these risks. If the frying oil contains high amounts of PUFA, the resulting risks are that much bigger. Therefore, the use of high oleic oils for frying are recently encouraged. Modified sunflower seeds have a reduced linoleic acid and increased oleic acid content. Thus high oleic sunflower oil has both higher oxidative stability and positive effects on health. Therefore the aim of our work, to determine the thermal stability of high oleic sunflower oil (omega 9) and to compare with the linoleic sunflower oil (omega 6) and refined olive oil which has also high oleic acid content.

Keywords: High oleic, linoleic, refined olive oil, thermal stability.

THE DIFFERENCES BETWEEN LINOLEIC AND HIGH-OLEIC SUNFLOWER OIL

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ABSTRACT

Linoleic type sunflower oil is mostly preferred oil in Turkey for different purposes, such as salads, meals, frying etc. However, in recent years, oleic acid type sunflower oil is more suitable and healthy for both frying and biodiesel has begun to spread, particularly in US, France and Spain. High oleic sunflower production should be expanded and encouraged due to many advantages. In the last few years, Trakya Birlik which is the biggest oil growers cooperative encourages to high oleic sunflower production in Turkey. Linoleic sunflower varieties are generally grown and processed industrially in Turkey. Linoleic acid reduces to the saturation and facilitates to the digestion and passes the blood. The greater amount of linoleic acid in the oil increases the oil quality. However, high linoleic acid content in sunflower oil affects to the industrial value. Linoleic sunflower oil usually use in salads, meals, margarines and shortenings. High oleic sunflower oil is used in generally spray oil in crackers, dried fruits, bakery products, frying, deep oil frying, roast process, salads and sauces, food supplements specialized for elders and child and as a mixture oil in margarine and mayonnaise. Except food industry, high oleic sunflower oil uses for cosmetic-paint industry and biodiesel production. The farming of oleic type sunflower is increasing and getting more important because the usage and consumer preference of oleic type sunflower oil are also increasing. While the US prefers to farm mid-oleic types contain maximum 80% oleic acid, Europe prefers to high oleic types contain more than 80%. Recently, hybrid seeds are used for sunflower farming and breeding programs have begun to high oleic seeds. However, it is still widely used linoleic sunflower for industrial purposes. Fatty acid composition not only affects to the industrial quality but also nutritional value is also affected at the same time. Fatty acid composition affects to the taste and chemical quality of oil. The phenolics in sunflower oil have effective role on taste aroma, oxidation level and rate.

Keywords: High oleic sunflower, linoleic acid, mid-oleic sunflower.

APPLICATION OF SUPERCRITICAL CARBON DIOXIDE FOR SUNFLOWER OIL EXTRACTION

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ABSTRACT

Sunflower is an important source for edible oil and thus commercially widely cultivated in order to produce vegetable oil in the world. Industrial extraction of sunflower seeds is commonly carried out through mechanical pressing followed by hexane extraction. However, this procedure might causes the production of undesirable residues, and also the oil can undergo oxidative transformations during the removal of the solvent. Therefore, oil quality is influenced negatively. In this study, a comprehensive review is presented on the researches and developments related to supercritical CO2 extraction of oil from sunflower seeds. Supercritical carbon dioxide (SC-CO2) extraction method is a promising potential alternative method for vegetable oil extraction to replace traditional techniques like mechanical pressing, organic solvent extraction. CO2 is the most commonly used solvent because of being non-toxic, non-flammable, non-explosive, cost-efficient, readily available, and easy to remove from the extracted materials. In SC-CO2 studies pressure and temperature during the extraction and recovery of the oil are important parameters that are considered. Moreover, the yield of oil is affected by the size and physical structure of the sunflower seeds.

Keywords: Supercritical Fuid, Extraction, Carbon dioxide, Sunflower oil

EFFECT OF ENZYMATIC INTERESTERIFICATION ON OXIDATIVE STABILITY OF SUNFLOWER OIL

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ABSTRACT

A preliminary study was undertaken to investigate the effect of enzymatic interesterification on the oxidative stability of sunflower oil. Immobilized lipase from *Rhizomucor mihei* was used to catalyze the interesterification of sunflower oil at 25±3°C. The immobilization procedure involves electrostatic complex formation between lipase and a highly branched polycationic polymer. Interesterification procedures were carried out in a continuous stirred batch type reactor. The comparison of oxidative stability based on simply peroxide value was carried out between virgin and enzymatic interesterified sunflower oils. The differences in peroxide values of virgin and interesterified oil samples were evaluated at the first day and 21th day of storage under accelerated conditions. The presented results of this study showed that the enzymatic interesterification decreased the peroxide value of sunflower oil. At the end of the 21th day of storage at 80°C, peroxide value of interesterified sunflower oil showed lower increase tendency than virgin oil.

Keywords: Interesterification, lipase, oxidative stability, sunflower oil

EFFECT OF THE DEEP-FAT FRYING PROCESS ON AROMA COMPOUNDS OF SUNFLOWER SEED OIL

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ABSTRACT

Sunflower seed oil is the fourth largest edible oil in the world and the consumption of it has been increasing due to the aromatic, nutritive and economic reasons. This vegetable oil is mainly used for cooking especially as frying oil. Deep-fat frying is a significant process of food preparation and gives a unique color and desirable flavor to food which is accepted by consumers. Through the process, a variability of chemical reactions result in the formation of extensive aromatic compounds. Likewise any other food product, oil quality is closely related to the aroma detected by consumers, and this attribute will have a great influence on its acceptance or rejection. Therefore, the present study explores the effects of the deep-fat frying process on aroma composition of the sunflower seed oil. The aroma compounds of 1, 5 and 10 times used oils to fry potatoes and non-fried oils were analyzed. Volatile components of the oils were extracted by using of the purge and trap technique with dichloromethane and analyzed by gas chromatography mass spectrometry (GC-MS). Results showed that total aldehydes content increased with the frying treatment due to the Strecker degradation. In this reaction, dicarbonyls or hydroxycarbonyl intermediates deaminate and decarboxylate amino acids to produce the corresponding Strecker aldehydes. Among the aldehyde compounds, (E,E)-2,4-decadienal, (E)-2-heptenal and hexanal were the major aroma compounds in all sunflower seed oils.

Key words: Sunflower, Deep-fat frying, Sunflower seed oil, aroma, GC-MS.

INTRODUCTION

Frying is the cooking of food in oil or another fat. Foods can be fried in a variety of fats and vegetable oils. These oils play a significant role in the food industry due to both their functional and nutritional features and their impact on taste, aroma and health. Refined sunflower oil, especially high-oleic, is very versatile and due to its neutral flavor and heat stability it can be consumed in many ways in the kitchen, such as frying and cooking. Sunflower (Helianthus annuus L.) is globally one of the most important oil crops and in

Turkey sunflower oil is commonly used for frying. Especially, deep frying is now the basis of a very large and expanding worldwide industry. During deep-fat frying, fats and oils are continuously or repeatedly heated at high temperatures (up to 190 °C) for prolonged periods of time in the presence of air. Under these conditions both thermal and oxidative reactions of the oils occur, leading to the formation of volatile and nonvolatile decomposition products (Chang et al., 1978). Prolonged deep-fat frying results in poor acceptability and nutritive value owing to the thermal and oxidative reactions in the frying oil. Together with the generation of long-lived bubbles and an increase in viscocity, frying oil begins to generate a noticeable odor attributable to the various volatile decomposition products of the thermally oxidized oil (Fujisaki et al., 2002, Tekelioğlu et al., 2008). Aroma is a main quality factor for edible vegetable oils as a characteristic parameter. During heat treatment by frying beside the aroma compounds formed which are very appreciated by consumers also other compounds which are not desirable get accumulated in the products; those compounds are formed by partial or total alteration of thermolabile nutrients present in food and in the frying oil (Ghidurus et al., 2011). The frying oil decomposition products as well as products formed from reactions between food components (proteins, cabohydrates) and oil constituents may adversely affect the flavor, color, nutritive value, and safety of the fried food (Takeoka et al., 1995; 1996).

Therefore, the aim of the present study was to evaluate the aromatic extracts obtained by purge and trap technique and then investigate the influence of frying process on aroma compounds. In present study, the aroma compounds of 1, 5 and 10 times used oils to fry potatoes and non-fried oils were analyzed.

MATERIALS AND METHODS

Materials

The water used in the study was purified by a Millipore-Q system (Millipore Corp., Saint-Quentin, France). Dichloromethane, 2-octanol, and sodium sulphate were obtained from Merck (Darmstadt, Germany). Dichloromethane was freshly distilled prior to use. Sunflower oil samples were obtained from a local producer in Adana city, Turkey.

Methods

Treatment of Frying Process

Before frying process, potatoes were hand peeled and then cut into strips (1 × 1 × 6 cm) with a stainless steel slicer. A fraction of 200 g of potato strips were used at each frying process. The potatoes were deep fried in sunflower oil using an electrical fryer (Felix FL 269, Turkey) at the 190 ºC for 10 minutes. Frying process was repeated 10 times and oil samples were obtained after 1, 5 and 10 times frying.

Extraction of the Aroma Compounds

The volatile compounds of sunflower oils were extracted by purge and trap system which consists in a source of nitrogen, controlled by a flow-meter. For the extraction, 10 g oil sample transferred into 20 mL vial then the sample was pre-incubated at the extraction temperature for 10 min. After purging process, the compounds retained in the cartridge were eluted with dichloromethane. After dehydration by anhydrous sodium sulphate, the pooled

organic extract was reduced to 5 mL in a Kuderna Danish concentrator fitted with a Snyder column at 40ºC (Supelco, St Quentin, France) and then to 0.5 mL under a gentle stream of nitrogen. Extracts were then stored at -20°C in a glass vial equipped with a Teflon-lined cap before analysis. Each sample was extracted in triplicate.

Analysis of Aroma Compounds

The GC system consisted of an Agilent 6890 chromatograph equipped with a flame ionization detector (FID) (Wilmington, DE) and an Agilent 5973N -mass selective detector (MSD). Aroma compounds were separated on a DB-Wax (30 m, 0.25 mm, 0.5 mm thickness; J&W Scientific, Folsom, CA) column. Retention indices of the compounds were calculated by using the retention data of a linear alkane series. After identification, the concentrations of aroma compounds were calculated by GC–FID according to the internal standard (2-octanol). The condition details of GC-MS and GC-FID were described in our previous study (Amanpour et al., 2015).

The GC system consisted of an Agilent 6890 chromatograph equipped with a flame ionization detector (FID) (Wilmington, DE) and an Agilent 5973-Network-mass selective detector (MSD) (DE, USA). Aroma compounds were separated on a DB-Wax (30 m length x 0.25 mm i.d. x 0.5μm thickness, J&W Scientific Folsom, CA , USA) column. A total of 3 μL of extract was injected in pulsed splitless (40 psi; 0.5 min) mode. Injector and FID detectors were set at 270°C and 280°C, respectively. The flow rate of carrier gas (helium) was 1.5 mL/min. The oven temperature of the DB-Wax column was first increased from 50° to 200°C at a rate of 5°C min-1 and then to 260°C at 8°C min-1 with a final holds at 260°C for 5 min. The same oven temperature programs were used for the MSD. The mass detector was operated in the electron impact mode at 70 eV. The GC-MS interface and ionization source temperature was set at 250°C and 180°C, respectively. Identification and quantification were performed in full scan mode with a mass/charge range of 30-300 amu at 2.0 scan s-1 scan rate. The compounds were identified by comparing their retention index and Wiley-6 and NIST-98 mass spectral libraries. Standard compounds were injected and analyzed under the same conditions. Retention indices of the compounds were calculated by using an n-alkane series. After identification the concentrations of aroma compounds were calculated according to internal standard (2-octanol) (Kesen et al, 2014).

RESULTS AND DISCUSSIONS

The volatile compounds identified in sunflower oils subjected to different number of frying process were presented in Table 1. Mean values (μg kg^{-1}) of the GC analyses of triplicate extractions were reported. As can be seen in Table 1, the total content of volatile compounds in the non-fried sunflower oil was the lowest and it increased gradually depending on the number of frying. The main reason of this could be taking place of oxidation, polymerization or thermal decomposition reactions in the frying oils used repeatedly at high temperatures. Besides, there are certain proofs that secondary oxidation products such as aldehydes and ketones, polar compounds, and acrolein which are formed in oxidized and degraded oils. Volatile compounds formed in frying oil include aldehydes, ketones, hydrocarbons, alcohols, acids, esters, and aromatic compounds (Chang et al., 1978). The amounts of volatile compounds of non-fried and 1, 5, 10 times fried oils were 2289, 3748, 5074 and 6992 μg kg^{-1}, respectively.

Results showed that total aldehydes content increased with the frying treatment due to the Strecker degradation. In this reaction, dicarbonyls or hydroxycarbonyl intermediates

deaminate and decarboxylate amino acids to produce the corresponding Strecker aldehydes. Among decomposition products during frying process, aldehydes are the most important because they are the most abundant (Frankel, 1985) and their thresholds are lower than those of other secondary products that characterize the flavor of fried foods and oils. As for identified aldehyde compounds in this study, (*E*,*E*)-2,4-decadienal, (*E*)-2-heptenal and hexanal were the major aroma compounds in all sunflower seed oils and their contents were increased due to number of frying process. The amounts of (*E*,*E*)-2,4-decadienal, (*E*)-2-heptenal and hexanal were found as 1448.0, 905.6 and 535.6 μg kg^{-1}, respectively. Volatile aldehydes are generated mainly from frying oil via β-scission of alkoxy radicals formed by the homolytic cleavage of FA hydroperoxides (Frankel, 1985).

Alcohols which were the other aroma compounds whose amount increased with the frying process. While their concentration in non-fried oil was 463.6 μg kg^{-1}, it reached 1417.5 μg kg^{-1} after the tenth frying treatment. The total concentration of pentanol, 2-nonanol, 2-methyl-2-butenol, 1-octen-3-ol and 1H-indole-3-ethanol increased with frying process.

Looking at the acid, lactone, terpene and phenol compounds, it has been observed that the amounts of such compounds decreased with the frying process.

When compared to literature, the different pattern of the aroma compounds of oils that subjected to frying process was determined. In previous studies, Doleschall et al. (2003) showed the aroma compounds of refined sunflower oil before frying and after the 3rd cycle. On the results of the refined oil small amount of hexanal, (*E*)-2-heptenal and nonanal have been observed, while the fried oil contains more types of aldehyde in larger amount. Chang et al. (1978) showed that the volatile products formed from corn oil and hydrogenated cottonseed oil during deep-fat frying. Other researchers have also studied the volatile constituents resulting from the thermal treatment of vegetable oils (Snyder et al., 1985; Macku and Shibamoto, 1991; Wu and Chen, 1992; Chung et al., 1993). These results suggest that frying process can effectively decrease or increase the amount of volatile compounds.

Table 1. Volatile Compounds of Sunflower Oil Under the Influence of Frying Process

		Number of frying process		
Aroma Compounds	**Non-fried**	**1**	**5**	**10**
Aldehydes				
Pentanal	-	142.0	215.4	344.9
Hexanal	227.4	287.1	304.2	535.6
(E)-2-Hexenal	-	45.8	71.7	124.0
(E)-2-Pentenal	-	-	-	36.5
(E)-2-Heptenal	101.1	766.1	796.8	905.6
Nonanal	-	148.9	175.2	197.1
(E)-2-Octenal	-	259.9	189.5	392.1
(E,E)-2,4-Heptadienal	-	-	128.6	33.3
(E)-2-Decenal	-	-	132.1	137.7
(E,E)-2,4-Nonadienal	-	-	21.5	24.8
(E,Z)-2,4-Decadienal	25.9	171.8	425.5	487.8
(E,E)-2,4-Decadienal	27.8	332.0	1181.1	1448.0
2-Heptedecenal	101.4	-	-	-
Total	**483.7**	**2153.6**	**3641.7**	**4667.3**
Alcohols				
3-penten-2-ol	129.2	94.3	82.5	72.7
Pentanol	65.0	51.8	123.1	226.3

4-Heptanol		44.4	26.4	25.4	16.7
2-Nonanol		-	-	-	92.6
5-Methyl-2-hexanol		121.1	79.5	71.7	-
2-Methyl-2-butenol		15.4	13.5	17.1	26.9
3-Octanol		30.2	-	-	-
1-Octen-3-ol		-	99.8	116.7	215.6
2-Phenylethanol		58.3	30.4	10.3	7.2
1H-Indole-3-ethanol		-	-	-	759.5
	Total	**463.6**	**395.7**	**446.8**	**1417.5**
Acids					
Pentanoic acid		24.8	43.2	-	-
Hexanoic acid		189.4	138.5	123.3	53.0
Heptanoic acid		45.4	73.2	-	-
Octanoic acid		111.6	92.5	38.2	21.5
Nonanoic acid		73.8	54.5	-	-
Decanoic acid		88.4	43.7	32.9	-
	Total	**533.3**	**445.5**	**194.4**	**74.5**
Ketones					
6-Methyl-2-heptanone		41.0	-	-	-
4-Nonanone		-	19.2	21.1	41.8
4-Hydroxy-4-methyl-2-pentanone		316.9	399.1	348.5	346.3
3-Nonen-2-one		-	48.3	78.7	87.2
2,7-Octanedione		-	37.2	49.4	62.7
	Total	**357.9**	**503.7**	**497.6**	**538.1**
Lactones					
5-Pentyl-2(3H)-furanone		7.9	-	-	-
5-Pentyl-2(5H)-furanone		40.5	26.8	-	-
	Total	**48.4**	**26.8**	**0.0**	**0.0**
Terpenes					
dL-Limonene		140.8	116.0	62.3	23.7
Linalool		-	-	16.2	14.2
	Total	**140.8**	**116.0**	**78.6**	**37.9**
Phenols					
Phenol		20.1	-	-	-
2,3-Dimethyl phenol		93.9	-	-	-
	Total	**114.0**	**0.0**	**0.0**	**0.0**
Esters					
n-Butyl acetate		146.8	107.3	94.8	65.7
Methyl palmitate		-	-	35.8	48.5
Methyl oleate		-	-	83.8	142.5
	Total	**146.8**	**107.3**	**214.4**	**256.7**
GENERAL TOTAL		**2288.6**	**3748.5**	**5073.5**	**6991.9**

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BIOPELLET PRODUCTION FROM WASTE MATERIALS OF THE SUNFLOWER IS A MAJOR INDUSTRIAL PLANT

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ABSTRACT

Sunflower is the most important industrial plant with oil content and consumption percent in Turkey. The highest production of sunflower with 44% is made in Thrace and a large content of waste materials (core-shell, sunflower bat etc.) are obtained after harvest and processing. These materials have alternative assessment opportunities. Untreated agricultural waste is generally used for heating directly. However, this method is not economical, efficient and suitable for environmental point. Harmful gases such as CO2 release during the combustion process occur. These waste materials leave to the field and return to the land again because of the difficulties and the lack of economic benefits with usage of heating material. However, it is possible that the waste materials can be converted into heating material, biopellet, is not harmful and has higher energy value. Biopellet is important heating material for farmer and sunflower oil industry. Farmers have a large amount of waste after sunflower harvest. Besides, high content of core-shell and solid material also get to stay in oil factories and cooperatives. Sunflower oil industry only annually produces 800 000 tons of solid waste in Turkey as a byproduct. Failure in evaluation of sunflower waste materials is too big to ignore is a serious economical loss. There are various studies about converting the sunflower waste materials after harvest and/or oil extraction. All of them say that biopellet production is valuable method for both environmental and economical. At the same time, the waste materials used as a heating material directly but inefficient combustion and excess content of volatiles were determined. All for these reason, biopellet is environmental friendly waste is a great need to improve fuel production. Although the ban, a significant amount of agricultural waste are burned in the field or using as fuel in homes in our country for each year. However, biopellet is a modern technic for heating offers integrated solutions for sustainable development in developed and industrial countries. Besides, it also serves the purpose of preventing climate change, erosion and efficiency, ecosystem health and loss of biodiversity. So, biopellet production is an ecological solution.

Key Words : Biopellet, core-shell, sunflower bat, sunflower waste.

FACTORS AFFECTING THE NUTRIENT COMPOSITION OF SUNFLOWER MEAL

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ABSTRACT

Sunflower (*Helianthus annus L.*) is a high oil-yielding seed crop cultivated worldwide that adapts very well to a wide range of climates. Sunflower seed meal is a by-product of the oil extraction of sunflowers and it is produced in large quantities. Sunflower meal (SFM) is mainly used as feed source that offer cheap, eco-friendly substrates for the animal nutrition. The meal is initially used as a protein complement in ruminant diets, and also monogastric animal rations in appropriate amounts. The chemical compositions of SFM have been extensively evaluated and it has been found that the chemical composition of SFM is varied greatly. The mean moisture and dry matter contents of SFM were reported as 9.0 % and 91.0 %, respectively. SFM is composed basically on lignocellulosic fiber and proteins. The content of crude protein in SFM ranges from 23.0 to 42.0 % and the crude fiber level varies between 13.0 % and 35.0 % depending on the extent of dehulling. The concentration of ether exracts in SFM varies from 0.50 to 13.0 % depending on the extraction process. The large variation of ether exract level was mainly related to the different extraction process. The differences in production methods, such as heating temperature, pressure and time during the process might lead to the changes in ether exract values. The different production techniques also caused the variation of the other chemical components of SFM. The content of phenolic compounds such as chlorogenic acid and caffeic acid in SFM ranges from 3 to 4 %. The average ash composition of sunflower meal was reported to be 6. 0 %. In conclusion, the processing techniques is one of the major factor affects the nutritional composition of SBM. Processing techniques are initially effective in the levels of ether exracts, the crude fiber levels and other nutrients therefrom. The variations of nutrient composition in SFM might result from dehulling process too. SFM composition can vary somewhat according to extrinsic factors such as genetic, seed varieties, climate and soil conditions. In addition, the chemical concentration of SFM is also affected in each plant and collecting typical samples in person and the analysis method used.

Key Words : Crude protein, crude fiber, nutrient composition, processing techniques, sunflower meal

EFFECT OF HIGH OLEIC SUNFLOWER OIL INCLUDING OLEOGEL ON THE TEXTURAL AND SENSORY PROPERTIES OF CAKE

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ABSTRACT

The existence of the relation between health and diet has motived people to consume food products with lower adverse health effects. As known consumption of excessive saturated fatty acid increases the risk of cardiovascular disease. Therefore, decreasing saturated fatty acid content of the food materials without damaging the quality of the food products is important issue in the food industry. When considering the importance of fats in the quality of the products, liquid oils are structured to transform them to solid fats. Oleogelation is one of the way which has been recently used for this aim. In the present study, probable usage of oleogels prepared from high oleic sunflower oil (HOSO) in the formulation of cake was investigated. For this aim three different oleogel formulations were studied: (i). 50 % cottonseed oil (CSO) + 25 % shortening + 25 % HOSO, (ii). 50 % HOSO + 50 % CSO and (iii) it is the same with second formulation however, this oil blend was oleogelled with dehydrated wax. Textural and sensorial properties of oleogel including cakes and control sample were investigated. Hardness, chewiness and gumminess values of the cakes prepared by oleogels were found to be higher than those of control sample. According to sensory analyses, the sample prepared from third formulation had the highest overall acceptability value. Wax type used in the formulation as well as oil types significantly affected textural and sensory properties of cakes. The findings of the present study highlighted that oleogels rich in unsaturated fatty acid content could be used in the cake formulation instead of shortening rich in saturated fatty acids.

Key Words : Oleogel, cake, high oleic sunflower oil, texture, sensory

XYLOSE PRODUCTION FROM PRETREATED SUNFLOWER STALKS

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ABSTRACT

Sunflower is an important oilseed plant. After harvesting of seed, stalks are left behind in the fields and they are usually left to rot or burned in the field with associated environmental risks. However, this biomass can serve as an abundant and renewable source for the soluble sugar, especially xylose. Xylose or wood sugar, is a member of aldopentoses. It is a sugar that is found in many edible seeds of the plant and located in the structure of plants, such as wood or straw. It can be obtained from hard and the soft woody plants or can be produced from agricultural waste that has a lignocellulosic nature. The most important use of xylose is xylitol production. Production of xylose from lignocellulosic materials are genarally carried out by dilute acid. The dilute acid hydrolysis is affordable, easy, fast and effective method for the production of xylose, but the hydrolysis requires corrosive chemicals, neutralization process and produce some undesirable compounds. The use of xylanase to hydrolyze xylan may be another alternative to acid hydrolysis of xylan for the release of xylose. The aim of this study was to evaluate *Trichoderma reesei* xylanase for obtaining xylose from autohydrolysis liquors of sunflower stalk. The effects of substrate concentrations and enzyme activity were investigated for the production of xylose. In order to obtain high xylose yield and selectivity, the optimization study was conducted by response surface methodology. Under the optimum condition, xylose yield and selectivity were found to be 86.4% and 9.2 g/g, respectively.

UTILIZATION OF SUNFLOWER STALKS

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ABSTRACT

Sunflower is an important and major oilseed crop in both Turkey and worldwide. After harvesting of seed, stalks are left behind in the fields and they are usually left to rot or burned in the field with associated environmental risks. In Turkey, around 2,500,000 tons of waste per year in the form of sunflower stalks are produced, which is a serious problem for farmers growing sunflower. So researchers focused on the assessment of using sunflower stalks and other agricultural waste for food and non-food purposes. These biomass can serve as an abundant and renewable source for the production of various chemical. The main component of sunflower stalk is cellulose that can be used in a wide range such as food, textile, cigarette industry such as film and sheet production. Cellulose derivatives has been used in rubber, paint, oil rigs, coatings, ink food, pharmaceutical industry. Hemicellulose and its fragmentation products have very wide range of applications in various fields. They have been used in the medical, cosmetic and food industry as absorbant, gelling agent, stabilizer, emulsifier, thickening. It can be converted to the film that can be used used for food packaging or fragmented to the oligomers that serve as prebiotics in functional food industry. Due to its amorphous structure, hemicellulose can be hydrolyzed by dilute acid to soluble sugar that can be used for the production of xylitol, lactic acid or ethanol. The aim of this study is to give detail information about the utulization of sunflower stalk.

NATURALLY BLEACHED VEGETABLE OIL, SHAPED BY ONE ALL-ROUND SOLUTION: TONSIL®

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ABSTRACT

Powerful against undesired odor, flavor and impurities from crude sunflower oil and other crude oils and fats, Clariant's TONSIL® bleaching earths have now been in use for more than 100 years. To meet today's growing global demand and ensure certified solutions for the scope of applications, we constantly carry out research into new products as well as into the rapid and flexible optimization of TONSIL® qualities in Europe, America and Asia. In many countries, TONSIL® has already become synonymous with activated bleaching earths, which we view as both a challenge and an obligation for the future.

Key Words : Bleaching Earth, Crude Oil, Sunflower Oil