Research Journal of Applied Sciences, Engineering and Technology 4(24): 5357-5360, 2012
ISSN: 2040-7467

Submitted: March 18, 2012 Accepted: April 14, 2012 Published: December 15, 2012

SATTVA-Statistical Affirmative Testing Tool for Various Adulterants

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Abstract: The aim of the study is to determine the adulterants in edible oil using Chromatography and authenticate the output using Statistical methods. Sattva in Sanskrit means Purity that is food should be healthy and unadulterated so as to strengthen the equilibrium of our human system. On consuming adulterated food it affects the digestive system, leads to metabolic disorders like diabetes, cardiac problems, also affects the human Mind and in turn the society. Food gives energy that includes carbohydrate, protein, fat, minerals and vitamins, of which fat constitutes 30% of the energy store. Edible oil is a source of fat that is consumed in large quantities across the world. In the proposed study, Sunflower oil adulteration with other cheaper oils detection using chromatography is identified. Using Principal component analysis the chromatographic results are authenticated.

Keywords: Adulteration, chromatography, edible oil, fat, food, principal component analysis

INTRODUCTION

Fat is a complex molecule and is the energy store (Durga Karthik *et al*., 2012) required for growth and synthesis of hormones for every human being. It is the slowest but an efficient form of energy for human being. Essential fatty acid is an important fat that cannot be synthesised by our body but should be taken along with food. These fatty acids are available from edible plant oils. Fat is of two types Unsaturated (Liquid at room temperature) and saturated (Solid at room temperature). Animals, plants and vegetable are the sources for these fats. Unsaturated fatty acid is good for health and can be classified as Monounsaturated [MUFA] and Polyunsaturated [PUFA] fatty acid. PUFA reduces the risk of cardiovascular disease and it is good when compared to MUFA.

Edible oil contains Fatty acids (El-Abassy *et al*., 2009; Priego *et al*., 2007), in the form of Triacylglycerol (Ruiz-Samblás *et al*., 2011; Piravi-vanak *et al*., 2009; Jeong and Dong-Sun, 2003), sterols, tocopherols etc. All these compositions can be analysed using chromatographic (Hye-Young *et al*., 2010; Aluyor *et al*., 2009) or spectroscopic devices (Jakab *et al*., 2002). As every oil has it is own compositions that differentiate it from the other oils, determination of composition by analysis proves to be a valuable method for detecting (Christophe *et al*., 2002) purity of the oils.

In India, traditionally Gingelly oil, Coconut oil, Ground nut oil are very common and recently Sunflower oil is used for cooking, pertaining to its Qualities like low LDL (Low Density Level) Cholesterol which is good for heart. In sunflower oil PUFA content is high and hence it is desirable to include in diet. As the demand increases, it is possible for the oil to be adulterated with cheaper oils like palm oil, castor oil, cotton seed oil. These adulterants in turn modify the natural qualities of the oil. Simple chemical analysis can be employed to detect adulteration but prediction of composition is not possible. Hence chromatographic or spectrometric analysis is required for exactly predicting the adulterants in a given sample. The signal data from these devices can be used to detect adulteration.

Table 1: Sunflower oil fatty acid composition

Fatty acid	(%)	Fatty acid	(%)
Palmitic acid	6.4	Stearic acid	1.3
Arachidic acid	0.4	Behenic acid	0.8
Oleic acid	21.3	Linoleic acid	66.2
Linolenic acid	<0.1		

To authenticate the data, it is further processed using Statistical tools such as PCA-Principal Component Analysis (Liliana *et al*., 2006) Anova etc. In the proposed method sunflower oil adulteration with cheaper palm oil is analysed using Gas Chromatography/Mass Spectroscopy (GC/MS) and to authenticate the result PCA is employed.

Sunflower oil analysis: Sunflower (*Helianthus annuus L.*) is the fastest growing oilseed (50% edible) crops in India that is light yellow in colour. The oil seed is also used as a nutritious meal for birds and animals, preparation of cosmetic and pharmaceutical products. The fatty acid composition for sunflower oil from The Merck Index is shown in Table 1

The sunflower oil contains more of linoleic acid and the absence of linolenic acid gives it a good aroma. Based

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on the fatty acid profile the sunflower oil is of four types:

- High Stearic/High Oleic
- High Oleic
- High Linoleic
- Nusun or Mid Oleic

MATERIALS AND METHODS

Using GC/MS, adulteration in sunflower oil is detected. As oils are non volatile it cannot be directly injected into the instrument and it has to be derivatized to make it volatile. The derivatization steps followed for oil analysis is given below, where fatty acids in oils are converted to corresponding methyl esters.

Steps for derivatization:

Step 1: Take 1 drop oil (sample to be tested), 1 mL Dichloromethane, 1 mL Sodium Methoxide. Shake the above content.

Step 2: Heat the above content in water bath at 45 degree for 3 min.

Step 3: Add 5 mL distilled water, cool it.

Step 4: Add 5 mL petroleumether, shake well and supernatant liquid that is formed is injected into GC/MS for analysis.

Gas Chromatography/Mass Spectrometry (GC/MS): GC/MS QP2010 + Shimadzu, Japan.

Program: GC: Polymethyl siloxene (100%) Column, 25 Micron, Pressure 100 kpa, Helium gas. Oven temperature maintained at: 230ºC, Inject at 250ºC.

Split ratio 1: 40, Total time: 30 min.

MS: Electron ionization, 40 to 350 mass/charge (m/z) ratio.

First pure sunflower oil and then palm oil is analysed to identify the fatty acid profiles. Then at various concentrations palm oil is mixed with the sunflower and the fatty acid profile is noted. For 1:1 concentration ratio of sunflower and palm oil, GC/MS fatty acid profile results are discussed below.

RESULTS AND DISCUSSION

Fatty acids are shown as methyl esters as esterification method is employed during derivatization. From the chromatogram results the fatty acid concentration is evaluated using the Eq. (1):

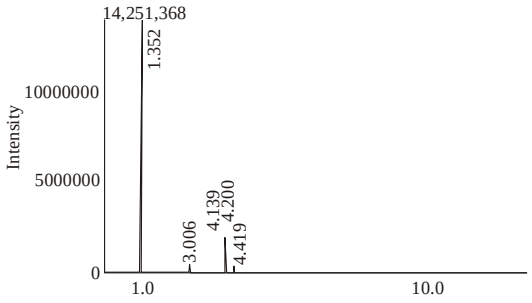
$$Fatty\ acid\ \% = \frac{Fatty\ acid\ area\ \%}{Total\ area\ \% - Solvent\ area\ \%} * 100$$

Table 2: Fatty acid concentration for sunflower oil

No	Retention time	% of fatty acid in oil	Name of the compound found	Name of the fatty acid
1	3.006	13.07	palmitic acid methyl ester	Palmitic acid
2	4.139	43.90	methyl linoleate	Linoleic acid
3	4.20	40.74	9 octo deconoic methyl ester	Oleic acid
4	4.419	2.29	methyl stereate	Stearic acid

Table 3: Fatty acid concentration in palm oil

No	Retention time	% of fatty acid in oil	Name of the compound found	Name of the fatty acid
1	2.237	1.195	tetra deconoic acid methyl ester	Myristic acid
2	3.008	39.96	hexa deconoic acid methyl ester	Palmitic acid
3	4.197	53.10	9 octodeconic acid methyl ester	Oleic acid
4	4.417	5.75	heptadeconic acid methyl ester	Margaric acid



Peak Report TIC

Peak #	R. Time	Area	Area%	Name
1	1.352	33956900	58.53	solvent
2	3.006	3145243	5.42	palmitic acid methyl ester
3	4.139	10560034	18.20	methyl linoleate
4	4.200	9797598	16.89	9-octo decanoic acid methyl ester
5	4.419	553107	0.95	methyhl stearate
		58012882	100.00	

Fig. 1: Chromatogram of pure sunflower oil fatty acid

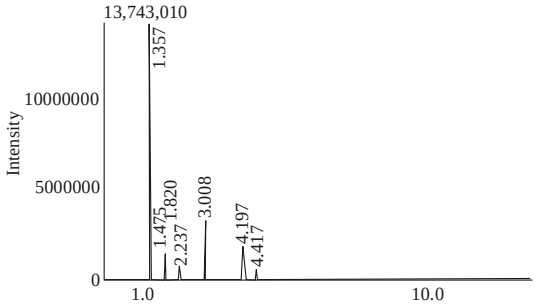
The fatty acid chromatogram report of pure sunflower oil is shown in Fig. 1.

Identification of fatty acid concentration % using Eq. (1) for pure sunflower oil is given below in Table 2:

It is seen that the Linoleic acid concentration is high (44%) and oleic acid is 40% for the sunflower oil sample. Other fatty acids like stearic and Palmitic acid contributes to (16%) of the total fatty acid concentration. Similarly the chromatogram of the palm oil sample is shown in Fig. 2:

The fatty acid concentration profile for palm oil is given in Table 3:

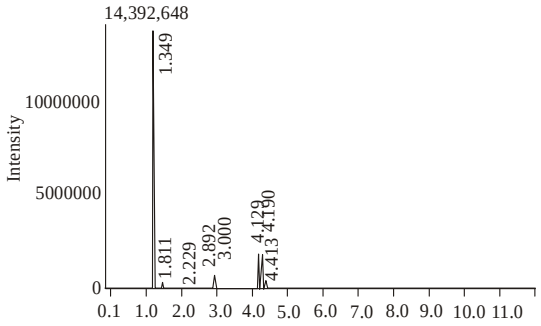
The oleic acid concentration is more (53%) followed by palmitic acid (40%), margaric acid (6%) and myristic



Peak Report TIC

Peak #	R. Time	Area	Area%	Name
1	1.357	36680986	69.21	solvent
2	1.475	55693	0.11	solvent
3	1.820	26515	0.05	tetra deconoic acid methyl ester
4	2.237	167973	0.32	tetra deconoic acid methyl ester
5	3.008	6401546	12.08	haxa deconoic acid methyl ester
6	4.197	8720396	16.45	9-octo deconoic acid methyl ester
7	4.417	944849	1.78	hepta deconoic acid methyl ester
		52997958	100.00	

Fig. 2: Chromatogram of palm oil fatty acids



Peak Report TIC

Peak #	R. Time	Area	Area%	Name
1	1.349	35359532	63.99	solvent
2	1.811	15349	0.03	nano decanoic acid methyl ester
3	2.229	81279	0.15	octo decanoic acid methyl ester
4	2.892	20395	0.04	--
5	3.000	4591811	8.31	haxa deconoic acid methyl ester
6	4.129	5154062	9.33	methyl linoleate
7	4.190	9369464	16.95	9-octo deconoic acid methyl ester
8	4.413	670010	1.21	methyl stearate
		55261902	100.00	

Fig. 3: Chromatogram of sunflower oil fatty acid

Table 4: Fatty acid concentration for sunflower oil with palm oil

No	Retention time	% of fatty acid in oil	Name of the compound found	Name of the fatty acid
1	1.811	0.083	nano decanoic methyl ester	-
2	3.000	23.07	0hexa decanoic acid methyl ester	Palmitic acid
3	4.129	25.902	methyl linoleate	Linoleic acid
4	4.190	47.057	9 octodecanic acid methyl ester	Oleic acid
5	4.413	3.775	octo decanoic methyl ester (methyl stereate)	Stearic acid

Table 5: PCA results for the data

	Initial eigen values			Extracted sum of squared loadings		
Component	Total	% of var	cumulative%	Total	% var	cumulative %
Raw 1	662.4	90.13	90.130	662.4	90.13	90.130
2	72.5	9.870	100			
Rescaled 1	662.4	90.13	90.130	1.793	89.64	89.649
2	72.5	9.870	100			

acid (2%) (Characteristic of Palm oil). Given in Fig. 3 is the fatty acid concentration analysed for sunflower oil and palm oil in 1:1 concentration ratio.

Fatty acid concentration profile for sunflower oil with palm oil is given below in the Table 4:

By analysing the weight of the fatty acid from the three tables, it is clear that the content varies from the pure sunflower oil and the adulterated oil. In pure sunflower oil palmitic acid (13%), Oleic acid (40%), Linoleic acid (44%) in the adulterant sample the concentration is 23, 47 and 25%, respectively. The rise in Palmitic acid concentration and the varying in concentration of other fatty acid itself shows that adulterations can be easily detected using chromatography. One new compound Nano Decanoic methyl ester is shown, where the fatty acid name is unknown. The chromatographic results are authenticated using statistical analysis that is performed on the data.

Principal component analysis: Principal Components Analysis is a method that reduces data dimensionality by performing a covariance analysis between factors. As such, it is suitable for data sets that have multiple dimensions. Principal component Analysis using SPSS is performed on the above data and the results are given below in Table 5.

From the above two it is seen that the variance is 90% for the first component of the sunflower oil and for adulterated oil (sunflower and palm oil). The palmitic acid content was taken as the first component for PCA analysis. The PCA results confirms the chemical report. In GC/MS report it is clearly seen that the palmitic acid content increased in percentage. In pure sunflower oil the palmitic acid is only 13% but in the adulterated oil at the same retentention time the palmitic acid is 23% that is

nearly very much high. This variance reported by PCA authenticates that the sample is adulterated.

CONCLUSION AND FUTURE WORK

Using GC/MS with PCA has found to give expected results and the methodology is easier and faster. A model can be constructed for detecting adulteration in sunflower oil by determining the fatty acid contents. The results shows that a neural model can be easily constructed by collecting the data from GC/MS and analysing it with statistical tools. Fatty acid composition is used for detecting other edible oil adulteration in sunflower oil similarly, detection of pesticides, heavy metals, plastic compounds and other toxic materials in sunflower oil are considered as an extension of this study.

ACKNOWLEDGMENT

My sincere thanks to the Managing Directors and Q/C Manager of M/S Lux Flavours, Chennai, India for suggestions and providing GC/MS Instrument to do the above research study.

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