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The Effects of Degumming and Neutralization on the Quality of Crude Sunflower and Soyabean Oils

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ABSTRACT: Degumming and neutralization processes are two essential stages carried out on crude edible oils in order to remove some unwanted impurities namely gums and free fatty acids. In this research study two popular less expensive commercial oils that are consumed to a great extent namely soyabean and sunflower seed oils are selected and subjected to both degumming and neutralization processes as recommended and described by the conventional methods. The effects of these two processes are investigated on both oils qualitatively and quantitatively. Minor changes were detected in fatty acid composition meaning that slight oxidation reactions have taken place during the operations. The acid values representing the fatty acid concentrations were reduced as the result of neutralization with the lye solution. The peroxide value that indicates the primary oxidation products has been increased due to the oxidation chain reactions producing hydroperoxide. The phosphorus, Iron and copper concentrations as well as the induction periods were reduced. The reduction in the induction period might be the result of phospholipid removals since they act as synergist with the tocopherols. The nonsaponifiable matter representing sterols, 4 methyl sterols, triterpene alcohols, tocopherols, hydrocarbons and some other components were reduced at the end of neutralization process along with the soap removal.

Keywords: *Degumming Process, Neutralization Process, Sunflower Seed Oil, Soyabean Oil.*

Introduction

Soyabean and sunflower seed oils might be considered as the two predominant vegetable oils in the industry due to a combination of factors namely nutritive, economical demand and large scale availability.

The refining operations include many stages that are carried out on the crude oils in order to improve the stability and remove the unwanted impurities that make the oil unfit for ideal behavior during cooking or frying practices (De Souta *et al*., 2007; Marrakchi *et al*., 2015; Jiang *et al*., 2014; Lamas *et al*., 2014). Degumming and neutralization stages are the two important stages carried out to remove phosphatides and free fatty acids. Degumming is the first step in refining processes. It removes phospholipids and gums. The presence of substantial amount of phospholipids can lead to the precipitation of gums in the carrying tanks, emulsion during refining operation or post cooking or frying practices, inactivation of catalyst if the oil is intended to be hydrogenated, production of oil with deeper

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color intensity and finally phospholipids can serve as the precursor of off-flavor such as fishy flavor (Erikson, 1995). Water and acid degumming methods are commonly applied in the industry. In water-degumming process, the hydratable phospholipids are removed from the oil by the treatment with water or steam usually at high temperatures. The resultant hydrated phospholipids become immiscible in the oil and are separated from the oil by centrifuging. During acid degumming, the hydratability of the phospholipids is increased by the addition of either phosphoric or citric acid (De Souta *et al*., 2007).

The gums produced after degumming process contain variable amounts of water, phospholipids, suspended matter and oil. Commercial lecithin can be obtained by removing water, oil and other components like glycolipids. Lecithin due to its emulsification properties might be employed in numerous applications in food and pharmaceutical industries among others (Penci *et al*., 2009).

The object of neutralization process is mainly the removal of free fatty acids that might cause problems during refining operations and post refining practices mainly frying. Substances namely traces of carbohydrates, proteins, resins, metals and some water soluble pigments might be removed when the oil is subjected to alkali neutralization.

The aim of this study is to investigate the effect of degumming and neutralization stages on the chemical composition and quality of soyabean and sunflower seed oils.

Materials and Methods

Crude, degummed and neutralized soyabean and sunflower seed oils were obtained from Behshahr oil industry.

Fatty acid composition of the oils were determined by formation of fatty acid methyl esters according to GhiassiTarzi *et al*. (2006) followed by the application of methyl esters to a HP-5890 gas chromatograph (Howlett-packed, palo AHO, Ca) equipped with cpsill 88 capillary column and flame ionization detector according to the standard AOCS method number, CE 1e-9 (Firestone, 1994). Acid value and free fatty acid concentrations were determined by the titration with potassium hydroxide using phenolphthalein as an indicator according to Ghavami *et al*. (2008). Peroxide value was determined according to AOCS standard method number cd 8-53 (Firestone, 1994). Iodine value was calculated according to the AOCS standard method number cd 1c-85 by employing the percentages of fatty acid composition. The oxidative stability of the oils was determined by the induction period measurements using Rancimat apparatus. The equipment measures the secondary oxidation products namely volatile acids that were released from the oxidized oil at 110°C in an air flow rate of 18-20 l/h according to Khorsandmanesh *et al*. (2012).

The phosphorous content representing the concentration of phospholipids and metals (Fe, Cu) were determined according to Ghavami *et al*. (2008).

The nonsaponifiable matter of the oils were isolated and quantified by the saponification of the oils with alcoholic potassium hydroxide followed by the extraction of the nonsaponifiables with diethylether according to Habibnia *et al*. (2012).

Results and Discussion

Table 1 presents the fatty acid composition of sunflower seed and soyabean oils as crude and degummed and neutralized. Sunflower seed oil is among oleic-linoleic acid group oils whereas soyabean oil is among linolenic acid group oils. The former has linoleic acid as the predominant fatty acid followed by oleic, palmitic and stearic acids as the major fatty acid constituents with minor quantities of others where the latter has linoleic acid as the predominant

fatty acid followed by oleic, palmitic, linolenic and stearic acids with some others minor constituents. The finding in this research work is in agreement with the results obtained by Farhoosh *et al*. (2009) and Penci *et al*. (2009). Both degumming and neutralization processes are carried out at atmospheric conditions and temperature of 60°C where a slight oxidation reaction might have occured. Slight reductions in the linoleic acid in both cases and linolenic acid in the case of soyabean oil might be due to this phenomena.

Table 2 presents the general characteristics of both oils prior and after degumming and neutralization processes.

Degumming has not caused any changes in the free fatty acids that have medium to long chain length since they are not soluble in water and not affected by degumming process. Due to the problems that free fatty acids create such as smoky condition during frying and lowering the smoke point, they are neutralized and removed as soap and the concentration has reached to an acceptable level in both cases. Further removals of free fatty acids are achieved at the later stage of refining, during deodorization process.

Peroxide value that represents the oxidative state of an oil, the primary oxidation product has not been reduced during degumming and neutralization processes. In fact increases in both cases are due to the fact that both processes are carried out at 60°C in the presence of oxygen where the formation of hydroperoxids is encouraged. The Iodine values have not been changed dramatically and the slight variation is due to the marginal changes in the fatty acid composition where the oxidation chain reactions have occurred.

The induction period representing the susceptibility of the oils to oxidation has been decreased for soyabean oil considerably while it has not been change for sunflower seed oil. This event might be due to the fact that phospholipids that are present in crude soyabean oil in a considerable quantity and act as synergist with the primary antioxidant namely tocopherols are removed substantially during degumming and neutralization processes.

The Iron and copper contents of both oils are reduced as the result of degumming and neutralization processes. This might be due to the removal of some phospholipids that might act as chelating agent and remove some of the chelated metals where in this case are copper and Iron.

Table 1. Fatty acid composition of oil samples (%)

Fatty acid	Crude sunflower seed oil	Degummed and neutralized sunflower seed oil	Crude soyabean oil	Degummed and neutralized soyabean oil
C14:0	0.07	0.07	0.10	0.09
C16:0	6.51	6.73	11.02	11.33
C16:1	0.07	0.08	0.10	0.08
C17:0	–	–	0.07	0.11
C17:1	–	–	0.04	0.05
C18:0	4.37	4.51	5.25	5.18
C18:1	25.97	26.33	22.33	22.63
C18:2	60.94	60.27	51.40	51.20
C18:3	0.14	0.11	7.94	7.79
C20:0	0.32	0.36	0.15	0.44
C20:1	0.15	0.14	0.46	0.57
C22:0	0.92	0.95	0.21	0.45
C24:0	0.27	0.30	0.20	0.08
Unknown	0.27	0.15	0.67	–

The phosphorus content representing the phospholipids concentration in the oil has been reduced to about 5 ppm, a figure that is the optimum concentration for this element. Phospholipids are generally desired compounds because of their synergistic activity that some might possess. However their presence in the oils is not desired due to precipitation, emulsion and inactivation of catalyst if the oil such as soyabean oil is intended to be hydrogenated and the last but not the least. The fishy flavor related to the flavor reversion of phospholipids. The nonsaponifiable matter of both oils are reduced as the result of neutralization process where some parts of the nonsaponifiable matter particularly if the compounds were present as esters and were hydrolised during the treatment with alkali were removed with the soap.

Fractionation of the nonsaponifiable matter on TLC are presented in Table 3 where it shows that the major constituents of the nonsaponifiable matter in both oils are sterols followed by tocopherols, triterpene alcohols, 4 methyl sterols and hydrocarbons in respective decreasing order. The work carried out by Ghavami (1982) indicated that the sterols are not affected by neutralization process while the tocopherols in the initial stages of refining; degumming and neutralization are slightly affected, but the major removal of this important and vital compounds occurred in deodorization stage. In both oil the predominant sterol was β-sitosterol followed by campesterol and stigmasterol.

Sunflower seed oil had Δ7-stigmasterol as a major fraction among the sterol fraction where this particular sterol might be accounted for the originality and identification of sunflower seed oil (Ghavami *et al*., 2008).

Table 3 shows the fractionation of the nonsaponifiable matter on TLC plate covered with silica Gel G type 60. The separation has occurred according to the polarity of the separated compounds and the colour intensity of the spots after spraying with acetic anhydride and sulfuric acid in ethanol. In the case of soyabean oil γ-tocopherol is the predominant tocopherol followed by δ and α tocopherols. In the case of sunflower seed oil α-tocopherals is the predominant tocopherol with traces of γ and δ tocopherols (Ghavami *et al*., 2008). As it has been explained by Ghavami *et al*. (2008) the later stage of refining, the deodorization, that is carried out at 260°C under vacuum removes almost the majority of the original tocopherals into distillated.

Table 2. Some quality characteristics of crude, Degummed and neutralized oil samples

	Samples			
	Crude sunflower seed oil	**Degummed & neutralized sunflower seed oil**	**Crude soyabean oil**	**Degummed & neutralized soyabean oil**
Nonsaponifiable matter (g/100g)) ± SD	1.18±0.032^{a}	0.94±0.043^{a}	1.47±0.038^{b}	1.13±0.012^{a}
Acid value (mg KOH/g) ± SD	2.03±0.192^{a}	0.13±0.028^{b}	1.92±0.042^{a}	0.15±0.311^{b}
Peroxide value (meq/kg) ± SD	5.97±0.021^{b}	8.90±0.012^{a}	3.990±0.0057^{c}	5.98±0.0196^{b}
Iodine value (g/100g)	128	127	129	129
Induction period(h) at 110°C	5.12^{b}	5.28^{b}	8.32^{a}	3.40^{c}
Phosphorous content (ppm)	50^{b}	5^{c}	180^{a}	6^{c}
Fe (ppm)	0.337^{a}	0.223^{b}	0.309^{a}	0.08^{c}
Cu (ppm)	0.015^{a}	0.008^{b}	0.013^{a}	0.002^{b}

Values in same row with same superscript are not significantly (P<0.05) different.

Table 3. Qualitative and quantitative fractionation of nonsaponifiable matter

Zones on TLC plate	Fractions	Rf value	Colour (under UV)	Composition in nonsaponifiable matter of sunflower seed oil (%)	Composition in nonsaponifiable matter of soyabean oil (%)
1	_	0.0-22.4	colorless	7	5
2	Sterols	22.4-31.8	yellow	40	49
3	4-methyl sterols	31.8-38.8	yellow	10	8
4	triterpene alcohols	38.8-43.5	yellow	15	15
5	tocopherols	43.5-64.7	violet	15	14
6	various	64.7-92.9	violet	8	4
7	Hydrocarbons	92.9-100	yellow	5	5

Conclusion

Soyabean and sunflower seed oil were subjected to degumming and neutralization processes where the unwanted components namely phospholipids and free fatty acids were removed. The induction period representing the stability of the oils toward oxidation was reduced. This is due to the removal of phospholipids that act as synergist in the presence of tocopherols.

Slight concentration of nonsaponifiable matters was lost with the soap removal as the result of neutralization. The removed sections might have been components that were esterified to the free sterols or other chemical compounds. Fatty acid compositions were not affected and the slight variation in the composition might be due to the slight oxidation of the unsaturated fatty acids during degumming and neutralization stages.

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