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**IMPACT OF PREPARATION PARAMETERS OF SOYBEAN ON PHOSPHATIDE,  
PEROXIDE, FREE FATTY ACID AND IRON AND COPPER IN RAW OIL IN  
INDUSTRY**

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**ABSTRACT**

Vegetable oil is a strategic product in Iran. Importation amount of this product is important and for the same reason the attempt for producing this raw oil inside of country is considerable in terms of competition with its imported counterpart in terms of production and quality. This research examined the impact of preparation stage of oil seed on quality of extracted raw oil with solvent at industrial scale with presenting details about devices performance and work conditions. Examined quality indexes in this study include phosphor, free fatty acids, and peroxide. Used Oil seeds is imported and from Brazil. The seeds after cleaning stage are crushed and cooked and prepared using flicker, expander and press devices. Solvent extraction (SE), Expander aid solvent extraction (ESE), Screw press (SP) and cake presses finally are transferred with solvent to extractor for oil extracting. The obtained oil is subjected to quality test by these three methods. Which comparing conducted test results on obtained oil samples that are prepared by above three methods, the obtained oil from preparation with SP method has indicated lower phosphatide and as a result lower refinery loss level. In comparison with quality of raw oil the method of ESE has demonstrated the best quality in terms of peroxide value and the SE method the best quality in terms of free fatty acid level. The significant difference of tests results has indicated the importance of controlling parameters of preparation stage for seed in oil extraction factory production lines. The results of this research are used in oil industry of Iran.

**Keywords: Raw Oil, Oil Seed Preparation, Phosphatide, Screw Press, Expander**

## INTRODUCTION

Soybean oil is one of the widespread vegetable oil containing considerable amount of unsaturated fatty acids. This oil contains alpha linoleic acid (omega 3), gamma linoleic and arachidonic (omega 6) oleic acid (omega 9) and is appropriate for human nutrition [1, 2]. In addition soybean oil contains palmitic and stearic saturated fatty acid [3]. The quality and quantity of raw oil is very important at grading time in terms of economy. One of most important factors that is examined in this section includes free fatty acids, oxidative stability, color and phosphatides [4-9].

Importance of usage from seeds preparation methods before oil extraction by solvent is owing to increasing efficacy and facilitating oil extraction from seed [10, 11]. The soybean oil contains high level of phosphatide and phosphor. And because phosphatides has negative influence on taste and color of the oil it should be separated from the oil. Phosphatide can be divided into two general category of hydrophilic and non-hydrophilic, where hydrophilic phosphatides can easily be separated by water degumming. In case of non-hydrophilic phosphatides separations more advanced processes is needed such as usage of acid for turning the phosphatides to hydrophilic type [12, 13].

The development of lipolysis is determined on the basis of free fatty acids level (with acid value). The oil with free fatty acids value higher than 1% is usually named as corrupted vegetable raw oil or animal fat. The only exception is the olive oil that even though has 3% free fatty acid it is still appropriate for direct usage [14]. The free fatty acids content is one of the signs of quality of warehousing and oil and fat production. Harm to seeds, degeneration of cell wall, collision of seeds to tough surface or other mechanical harms are among factors that leads in increase of free fatty acids in the oil. The high humidity of seed make it easy for lipase enzyme activity and it is worthy to mention that this enzyme is more active in sprouted seeds. The smoke point in oils depends on free fatty acid level in the first stage, owing to the fact that free fatty acids are more evaporating than other similar triglyceride [15].

The conventional method of soybean oil extraction is usage of organic solvent of hexane and thereby 99% of oil of seed is extracted and just 1% of oil remains in that [11].

Screw press is the second way of oil extraction. This method is one of seed preparation methods before oil extraction with solvent. In this way the remaining oil in screw press is 4 to 7 %. Screw press

works with low temperature and has appropriate cake quality but its production capacity is fairly low [11]. One of other seed preparation ways before extraction by solvent is usage of expander. One of important reason of using expander includes: inactivating non-nutritious factors of oilseeds for their usage in cattle feed, extracting oil from seed mechanically before extraction by solvent, facilitating extraction process by solvent for the reason of destroying spherosomes that leads in releasing oil, inactivating fat destroying enzymes and food production that their available oil content is eliminated to some extent. Expanders increase capacity of extraction devices by solvent due to volume reduction [16]. Among important factors of quality of raw oil that effects on production loss in oil refinery stage include peroxide, phosphatides, free fatty acid percentage and oil color. Regarding direct dependency parameters in oil extractions the type of extraction oil method and operatory conditional influence greatly on final product quality.

## MATERIAL AND METHODS

### Used materials

Used oilseeds in this research are imported soybean from Brazil. Due to importance of input seed quality into product line in regulations of preparation parameters, at first seeds were subjected to analysis of

determination of external substances percentage, humidity, oil percentage and acidity level. All analysis methods are in accordance with AOCS method 2009 or Iran national standard.

The used solvent for chemical oil extraction in extractor is hexane from permissible type for usage in food industry that its analysis is determined by Gas chromatography (GC).

All used chemicals in this research are provided from Germany Merk company agency. For measuring humidity the psychrometer scale from Satrius company model MA35 is used. The thickness measurement is taken place by digital micrometer device in this research for phosphor content measuring the spectrophotometer Shimadzu device UV-1800 is used.

### The process and method of research condition

Winnowing and cooking of seed: this reseach is conducted practically in one of domestic oil extracting factories and in industrial scale. Soybeans after sustaining winnowing stages are transferred to cracker device equipped with four-grooved roller. The distance between grooves is almost 3.3 to 7 mm and 7 grooves per inch. The seeds pass through four rollers and the seeds are divided into three or four parts.

The cooking operations are carried out in five story vertical type cooker device during

35 to 45 minutes. The cracked seed humidity before entering to flaker is 10 to 12%.

### **Preparation with flaker**

The cracked and cooked soybeans are transferred from cooker to flaker, the seeds are turned into flakes with thickness of 0.2 to 0.3 mm by flaker device with hydraulic pressure between rollers. The thickness of flakes is sampled in three parts of flaker for making sure for evenness of thickness and is measures by digital micrometer.

### **Preparation with expander**

Soybean flakes are transferred to expander of single screw type by conveyor belt that works in lower temperature and pressure. Soybean flakes are subjected to pressure in expander and are turned into elastic and cohesive substances and are cooked up to 112.8 degree centigrade and humidity of 10 to 13 percent.

The output soybean from expander has porous structure that is called collet and it enters in chiller and dryer and its humidity is reduced to 7%.

### **Preparation with press**

Flakes are transmitted to pressure cooker by conveyor belt. The used press cooker is four story type and the output flakes humidity is considered by 1 to 4 percent. After cooking in cooker the flakers are entered into press.

### **Extracting by solvent**

Oil extraction by solvent from prepared flakes, collets and cake presses:

In subsequent stage flakes, collets and press cakes are subjected to oil extraction process by hexane solvent separately.

The extractor (solvent extraction device) is continuous type, horizontal and fabricated by DeSmet with inconsistent flow and work capacity of 350 ton per day. This device has 8 solvent showering spray on materials bed and 14 misella internal collector hopper. Height of ingredient on the meshes is adjusted as 1400 mm.

Misella (combination of Hexane and oil) finally after passing through economizer for exerting necessary temperature for solvent separation from misella passes through three hitter that based on naming of DeSmet company are called 18, 21 and final evaporator and after complete separation of solvent are chilled till 40 degree centigrade.

The work condition of extractor as well as heaters is considered as equal for all three conducted test. Finally for each one of tests for preventing desired oil mixture with existing oil in storage tank, the obtained oil is sampled in an appropriate sample vessel according to test defined specifications from sampling faucet before oil everyday storage tank. The phosphor value determination test in raw oil with method of AOCS CA 12-55, and Free fatty acids amount determination with method of AOCS CA 5a-401 and

peroxide by AOCS cd 8-53 (03) are conducted.

### Statistical analysis

All experiments are taken place in three repetition, and data analysis by Spss software version 17 and comparing data averaged in 0.95 reliability level is carried out with LSD and Duncan.

## RESULTS AND DISCUSSION

Determination of external substances percentage, humidity, oil percentage and acidity level was done in accordance with AOCS method 2009 or Iran national standard and the results are shown in **Table 1**

Solvents were used for chemical oil extraction in extractor is hexane from permissible type for usage in food industry that its analysis was done by Gas chromatography (GC) and the results are shown in **Table 2**.

**Table 3 shows the** operatory conditions of all used production devices.

### Peroxide and free fatty acids value:

The results of acidity and peroxide test are shown in **Table 4**. At level of confidence of 0.95 comparing peroxide value (based on milliequivalent per sample oil kilogram) shows significant difference in different method of extraction. In the same way the free fat acids value (based on sodium oleate) shows significant difference in results of

different methods of extraction at level of confidence of 0.95.

In **Table 5** results of determining phosphor and phosphatide content in obtained oil from different methods of soybeans preparation is presented.

Comparing heavy metals content of iron and copper in oil. Iron and copper content is reported in **Table 6**.

The type of oil extraction influences on quality of obtained oil and cake. The significance differences in peroxide and acidity value of prepared obtained oil with different methods of preparation suggests the influence of production conditions on speed and amount of inactivation of lipase and lipoxygenases enzyme available in seed that due to destroying cell wall it stands beside its substrate namely seed oil and cause its destruction [17]. In comparing different types of preparation methods, in screw press due to applying low temperature lower than enzymes inactivation temperature and lower humidity level the enzymes are not inactivated completely. This relative inactivation of enzymes can influence negatively on oil quality over preparation time. In extracted oil from seed by expander preparation method owing to the fact that all seeds do not sustain equal thermal curing, the destructive enzymes among them have longer time for contacting with their substrate and leads in higher level

of peroxide than flaker method. Another effective parameter in peroxide value and free fatty acid content is soybean warehousing and production conditions. When humidity and thermal conditions over seed warehousing is inappropriate the lipase and lipoxygenases can be activated. Regarding equality of used seeds in the research and equality of preparation condition up until flaker stage the influence of these factors on results of different preparation methods is equal.

In solvent extraction system in the stage of separating hexane from misella steam spraying may be the reason for engendering appropriate thermal and humidity conditions and it leads in accelerating peroxide formation [18].

Seeds humidity level, temperature and time of production influence directly on phospholipase D enzyme activity and turning hydrophilic phosphatide to non-hydrophilic phosphatide [19].

When the soy protein is denatured and its structure unfolds, it is subjected to hydrophilic compound, the phospholipids reacts by hydrophilic heads and the cake is remained connected and resist against separation during oil extraction. Therefore hydrophilic phosphatides are expelled with cake and the result oil contains less amount of phosphor. Phosphatide amount and thereby the loss amount in water degumming stage in the obtained oil is more in ESE method and less in SP method .

As it can be observed there is no significant difference between usage from screw press and expander and flaker in copper content. Though these results shows lower level of iron and copper in EE.

Furthermore contacting hexane with steel carbon metals that leads in B degree corrosion can be another factor for increase of iron content in final product.

**Table 1: Analysis of preliminary seeds**

The seed/ analysis and test method	External substances percentage National standard 661	Humidity percentage AOCS Ac2-41	Seed oil percentage AOCS AC3-44	Free fatty acids percentage AOCS Ca 5a-401
Brazilian soybean	0.5	8.74±0.036	20.24 ±0.25	0.746±0.015

**Table 2: Pure percentage of used hexane in oil extracting by solvent**

Number	Compound name	The under peak level in GC spectrum (%)
1	N- Hexane	58.1585
2	Methylcyclopentane	21.215337
3	Methylepentane	9.9427
4	2- methylpentane	9.0472
5	Other cases	1.636263

Table 3: Used production devices operatory conditions

Device	parameter	unit	value	
cooker	Cooker stories temperature	First story	°C	68
		Second story	°C	76
		Third story	°C	87
		Fourth story	°C	97
		Fifth story	°C	100
		Sixth story	°C	100
		Seventh story	°C	100
	Steam pressure	Bar	7-10	
	Retention time of material in cooker	Minute	45 minute	
	Humidity of input seed to cooker	Percent	8	
Humidity of output seed from cooker	Percent	12		
flaker	Hydraulic pressure	Bar	38	
	Current	Ampere	44	
	Flakes thickness	MI meter	0.25	
	Flakes humidity	Percent	8	
expander	Injection steam	Bar	4	
	output collect temperature from expander	°C	110-120	
	Output collect temperature from cooler dryer	°C	40-60	
	Output collect temperature from cooler	Percent	6-8	
Press cooker and cooker	Applied temperature to flakes in cooker	°C	120-115	
	Heating time in cooker section	Minute	45 minutes	
	Output flakes humidity from cooker press	Percent	0.4-1	
	Input load temperature to press	°C	Near 117	
	Applied temperature to soybeans at press	°C	121-148	
	Applied pressure to soybeans	Psi	15000-20000	
	Remaining oil percentage at press cake	Percent	12 to 14%	
extractor	material bed height on extractor	Mm	1400	
	Extractor internal vacuum	mmH2o	-1	
	Extractor internal temperature	°C	50	
	Extractor sprays temperature	°C	52-55	
	Number of extractor meshes pores	Per In2	28	
	Extractor current	A	6	
	input voltage of extractor	V	390	
Solvent separation at heaters	heater 18 temperature	°C	94	
	Heater 18 vacuum	mmHg	380	
	21 heater temperature	°C	105	
	21 heater vacuum	mmHg	460	
	Final evaporator temperature	°C	110	
	Output oil temperature at cooler	°C	40	

Table 4: The impact of extraction method on free fatty acids and peroxide amount

Measured parameter	SP	SE	ESE	unit
Peroxide value	0.98±0.1	.67±0	0.24±0.06	Meg/kg
Free fatty acids	1.12±0.01	0.93±0	1±0.2	%

Table 5: Test result of determining phosphor content on obtained oil by different methods of oil extraction

Measured parameter	Extraction type			unit
	SP	SE	ESE	
Peroxide value	448±7.63	552±2.5	1080±2	Ppm
Free fatty acids	1.34	1.65	3.24	%

Table 6: Comparing copper and iron content in extracted oil by different methods

Measured parameter	Content	Extraction type		
		SP	SE	ESE
Iron	Ppm	4c	A2.4	B2.5
copper	ppm	A0.05	A0.05	A0.05

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