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Effect of Different Parameters on Removal and Quality of Soybean Lecithin

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Abstract: With regard to study the effective factors on removal and quality of lecithin extracted from soybean oil and the residual amount of phosphatids in degummed oil, this study was arranged. Crude oil recovered from soybean which had been processed by conventional solvent extraction and reached to a phosphorus content of 454 ppm, was used for this purpose. Treatment were carried out under different concentrations of phosphoric acid (0, 0.05, 0.1, 0.2, 0.5, 2) and different percents of water (0.5, 1, 2, 5, 3, 4) at different temperatures (25, 50, 60, 75, 90°C) and with different stirring times (5,10, 20, 40, 60 min). The highest phosphatid recovery was obtained with 3% (v v^{-1}) water at 75°C with stirring time of 20 min. Phosphoric acid adding decreased the phosphatid residue in degummed oil but the quality of lecithin was reduced. However addition of phosphoric acid lower than 0.05% (v v^{-1}) at 60°C resulted the same recovery efficiency without important effect on quality of lecithin.

Key words: Soybean oil, lecithin, phosphatid, extraction

INTRODUCTION

Nowadays study of foods with natural origin has allotted a special place to itself compared to that of artificial origin foodstuff and valuable results are also obtained in this respect. Lecithin is an example of such materials that its natural origin has led to increase the popularity of those products that utilize of it instead of synthetic compounds (Wendel, 2000). During the recent century, human has recognized the value of lecithin and according the researches carried out, the highly important and essential roles of lecithin have been studied both in industry and various nutritional and therapeutical areas as well. Improving of lecithin quality and its yield extraction have been always under focus of producers in various food industries (Wu and Wang, 2003; Arvanitoyannis, 2003; Orthoefer and List, 2006).

Presence of phospholipids or phosphatids, or in other words gums, in vegetable oil causes a higher oil loss in neutralization stage and on the other hand generates numerous difficulties while oil being transported from tanks and more even can obstruct the pipes completely. Therefore, by simple degumming not only the above problems would be prevented but also the separated gum could be sold as commercial lecithin in the market (Bernardini, 1985; Karwezyk, 1996; Wendel, 1984). The basis of extraction of phosphatids relied on this principle that phospholipids become swollen in treating with hydrating substances and is separated in form of precipitation. For this, the hydrating material must be mixed with oil and they must be extracted after sedimentation of phosphatids. Thus insolubility of hydrated phosphatids in oil and the higher specific gravity lead to a simple separation of them (Wiedermann, 1981; Haraldsson, 1983).

Water has been used as a traditional hydrating substance for separating hydratable phosphatids since the first years of industrial vegetable oil processing. About 90% of phosphatids of soybean oil, extracted from good quality seeds, are hydratable phosphatids. During appropriate water induced degumming, about 95-98% of phosphatids, which mainly consisted of alpha lipoid, are separated (List, 1981). The amount of added water for hydration of phosphatids is very critical. If water addition is lower than needed amount, hydration would not be done appropriately and if the amount of water is higher than of its optimal value, it results in high oil loss and decreasing the percent of phosphatids in extracted gums. In some oil extraction plants, the gums are hydrated using condensed vapor of misselate solvent extraction section. In this case, the amount of added water should be controlled carefully due to direct vaporization and often it is preferred that warm water being used instead (Shahidi, 2005). Among other hydrating agents, some inorganic acids such as phosphoric acid, boric acid and several organic acids like propionic acid, succinic acid, citric acid, acetic acid and oxalic acid could be mentioned (Smiles *et al.*, 1989). The extraction efficiency, purity degree and color quality of lecithin could be affected by selecting each of these compounds. Many other factors have also been reported by researchers to have influences on the phosphatids hydration such as, amount of hydrating substance, hydratation temperature, stirring time and speed, centrifugation time and some other minor parameters (Shahidi, 2005).

The aim of this study is not only to study the effect of these parameters on the phosphatids hydration yield but also to consider their influences on the quality of lecithin produced and degummed oil.

MATERIALS AND METHODS

Soybean crude oil with a phosphorus content of 454 ppm was used throughout of this work. The phosphatid content normally could be calculated by multiplying of phosphorus content by 30 (Racicot and Handal, 1983). All other chemical materials such as phosphoric acid, zinc oxide, sodium molybdate, chloridric acid, potassium dihydrogen phosphate, potassium hydroxide, sulfuric acid and hydrazine sulfate were in analytical grade.

The research carried out with the following procedure: water and phosphoric acid have been added in different ratios as hydrating agents and influence of factors such as temperature, mixing time duration of oil and hydrating agent as well as mixer speed (rpm) in lecithin extraction efficiency have been studied. In each experiment, after hydration of phosphatids, the obtained mixture conducted to a centrifuge (Optimal 1BHG 602, USA) which separated it in the two phases at 3000 rpm and 30 min. The lower phase contains extracted lecithin and upper phase is degummed oil.

The optimal conditions should be identified based on the minimal residual phosphorus content in the degummed oil and the quality of separated lecithin especially its apparent color. For this purpose, samples of 100 g crude oil were prepared.

At first, for determining optimal mixing time of soybean crude oil with hydrating material, experiments were carried out by using of water alone or diluted phosphoric acid as hydrating agents. Other parameters kept constant. Mixing time variations were 5, 10, 20, 40 and 60 min. After phase separation, phosphorus content of degummed oil and color quality of lecithin were determined.

In the next step, different percents of water (0.5, 1, 2, 3 and 4%) and various percents of water and phosphoric acid in combination [(water, 3% plus phosphoric acid, 0.0%); (water, 2.95% plus phosphoric acid, 0.05%); (water, 2.9% plus phosphoric acid, 0.1%); (water, 2.8% plus phosphoric acid, 0.2%); (water, 2.5% plus phosphoric acid, 0.5%); and finally (water, 1% plus phosphoric acid, 2%)] were applied. Each of these steps performed under different temperatures (25, 50, 60, 75 and 90°C), for 20 min of stirring and with 3 repetition for each.

RESULTS AND DISCUSSION

Influences of four factors i.e., water adding, time of stirring, temperature and increase in phosphoric acid concentration on lecithin recovering from soybean crude oil were studied and following results were obtained:

Effect of amount of water addition: As seen in Fig. 1, with increasing the amount of water added to crude oil, the lecithin recovering efficiency increased and reached to a maximum value of about 73% at water percent addition of 3. However, more increasing of the amount of added water subjected to a lesser efficiency. In fact, if the water addition exceeds from a certain limit, the water and oil emulsion could be formed and it would decrease the efficiency of lecithin recovering.

A statistical analysis also performed for better interpretation of results. The analysis showed that there is a significant difference between 0.5 and 1% of water versus 2, 3 and 4% (Table 1). Also there is no significant difference between 2, 3 and 4% of water ($p<0.05$).

Regarding these results, it should be mentioned that despite some reports (Shahidi, 2005; Indira *et al*., 2000) which suggest a water addition equal to phosphatid content of the oil, the optimal value needs to be determined experimentally for any crude oil (Arvantoyannis, 2003; Orthoefer and List, 2006).

Influence of stirring time: As seen in Fig. 2, By increasing mixing time and as a result prolonging reaction time, it could be expected that the phosphatid removal efficiency will also be increased. However, by considering the adverse effects of temperature on color of lecithin over time, an optimal stirring time should be defined.

Table 1: Statistical analysis of water effect on the phosphatid residue in oil

Water content (%)	Residual phosphatid (%)	Std deviation	F (p-value)	Comparison of water content*
0.5	1.1933	4.509×10^{-2}		0.5 and 1 versus
1	0.7167	2.517×10^{-2}		all others.
2	0.4333	2.082×10^{-2}		there is no
3	0.3667	1.032×10^{-2}	450.982	significant differences
4	0.4867	1.528Î 10^{-2}	$p<0.001$	between 2, 3 and 4

*One way analysis of variance

Table 2: Effect of stirring time on the phosphatid removal performance in presence and absence of phosphoric acid

Water content (%)	Phosphoric acid (%)	Run No.	Stirring time (min)	Residual phosphatid (%)	Std. Deviation	F (p-value)	Sig*
3	0	1	5	0.4933	1.528×10^{-2}	33.598	1 versus all others.
3	0	2	10	0.4133	1.528×10^{-2}	p<0.001	2 versus 1 and 5.
3	0	3	20	0.3667	2.082×10^{-2}		There are no
3	0	4	40	0.3500	3×10^{-2}		significant
3	0	5	60	0.3100	2×10^{-2}		difference between 3,4 and 5.
2.9	0.1	1	5	0.2933	1.528×10^{-2}	8.566	
2.9	0.1	2	10	0.26	0.01	p<0.003	1,2 and 3 versus
2.9	0.1	3	20	0.25	3×10^{-2}		4 and 5.
2.9	0.1	4	40	0.24	3.6×10^{-2}		There is no significant
2.9	0.1	5	60	0.16	5.19×10^{-2}		difference between 4 and 5.

*Multiple comparisons, dependent variable: phosphatid residue, scheffe

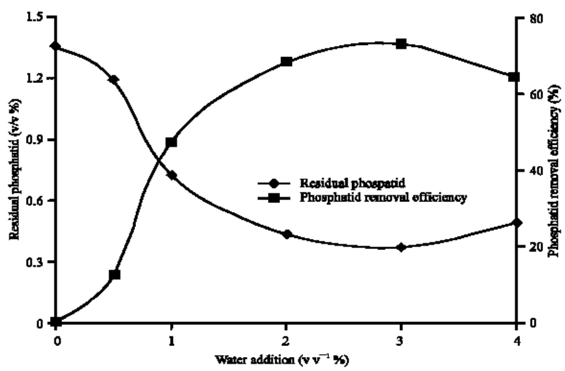

Fig. 1: The effect of water addition percent on the phosphatids recovery from soybean crude oil. (mixing time = 20 min, T = 75°C)

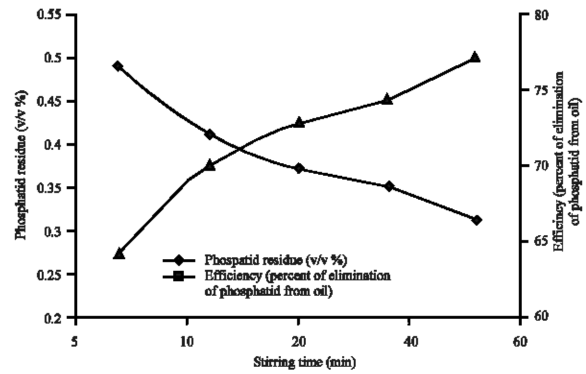

Fig. 2: The effect of stiring time on the phosphatids recovery from soybean crude oil, water = 3% (v v^{-1}), T = 75°C

The results showed that there is a significant difference between various applied methods in terms of time of stirring (Table 2). It is obvious that the hydration rate of phosphatids is very high in such a way that in water system within the first 5 min. about 64% of gums are separated and after 10 and 20 min of stirring, the efficiencies increased to 69 and 73%, respectively and maintenance of stirring until 1 h resulted in to only about 4% increasing of phosphatids elimination. Statistical analysis (Table 3) showed in this system there were significant differences between 5 and 10 min with 60 min but there were no significant differences between 10, 20 and 40 min stirring times ($p<0.05$). However, with consideration the point that lecithin is a heat sensitive substance with dark discoloration therefore the stirring time of 60 min in spite of generating greater amount of phosphorus elimination is not suitable for lecithin recovering.

In the case that we utilize a mixture of water and phosphoric acid instead of water for a lecithin recovering method, a relatively high speed process with a better efficiency will be followed i.e. within the first 5 min about 78.7% of phosphatids are eliminated and at 10, 20, 40 and 60 min after stating lecithin recovering, the efficiencies increase to 80.9, 81.6, 82.35 and 88.2%, respectively. However when phosphoric acid presents in the system, prolongation of stirring time affects an undesirable effect on lecithin color. Statistical analysis showed the significant differences between 5, 10 and 20 with 40 and 60 min stirring times ($p<0.05$). Hence, in this study a 20 min of stirring time assigned for lecithin recovering seems to be suitable for both water and combination of water and acid methods which is near to the mixing time reported by List (1981).

Effect of temperature: By increasing temperature, the percent of phosphatids elimination from soyabean crude oil should be increased. Our results showed that in water system at 25°C, the efficiency of lecithin production is only 33% and by increasing temperature to 75°C, the phosphatids elimination percent gradually increases but at 90°C a significant decrease appears (Table 4). At this temperature the apparent color of lecithin turns to deeply dark. When with increasing temperature the phosphoric

Table 3: Statistical parameters in the analysis of stirring time effects on the phosphatid residue in soybean crude oil

Source	Degree of freedom	Sum of squares	Mean square	F	p*-value
Corrected model	9	0.247	0.02742	42.403	P<0.001
intercept	1	2.958	2.958	4574.041	P<0.001
WAT-Phos	1	0.158	0.158	244.969	P<0.001
St.time	4	0.08029	0.02007	31.039	P<0.001
WAT-Phos st.time	4	0.008087	0.002022	3.126	P<0.038 **
Error	20	0.01293	0.0006467		
Total	30	3.218			
Corrected total	29	0.260			

*Univariate analysis of variance, Multiple comparison. ** Significant difference

Table 4: Comparison of phosphatid residue at different temperatures and in different systems

Water-phosphoric acid	Run	Temp	Residual phosphatid (%)	Std. deviation	F	p-value	Sig *
	1	25	0.9067	0.03055			
	2	50	0.5367	0.04163			1 versus 2,3,4,5
3-0	3	60	0.5067	0.01528	172.093	p<0.001	
	4	75	0.3667	0.02082			2,3,5 versus 4
	5	90	0.5067	0.01528			
	1	25	0.7467	0.07767			
	2	50	0.4167	0.04163			
2.95-0.05	3	60	0.37	0.1732	34.539	p<0.001	1 versus 2,3,4,5
	4	75	0.36	0.5292			
	5	90	0.4067	0.02309			
	1	25	0.75	0.06			
	2	50	0.3833	0.1002			
2.9-0.1	3	60	0.2533	0.03512	36.449	p<0.001	1 versus 2,3,4,5
	4	75	0.31	0.02			
	5	90	0.3967	0.01528			
	1	25	0.3067	0.075			
	2	50	0.3	0.01			
2.8-0.2	3	60	0.28	0.01	6.047	p<0.01	3,4 versus 5
	4	75	0.2633	0.01528			
	5	90	0.41	0.04583			
	1	25	0.4067	0.02082			
	2	50	0.5567	0.1002			
2.5- 0.5	3	60	0.5	0.05568	9.358	p<0.002	1,3 versus 5
	4	75	0.5167	0.01155			
	5	90	0.6633	0.01528			
	1	25	1.2067	0.04726			
	2	50	1.1733	0.7572			
1-2	3	60	0.49	0.02	172.742	p<0.001	1,2 versus 3,4,5
	4	75	0.5633	0.2517			3, 4 versus 5
	5	90	0.8833	0.2517			

*Analysis of variance-Multiple comparisons

acid percents also increased, the higher efficiency of lecithin production in such a way that by adding 0.1% of phosphoric acid at 60°C and adding 0.2% phosphoric acid at 75°C the highest efficiency is obtained (81.6 and 80.9%, respectively).

Nevertheless, the lecithin color becomes dark at temperatures higher than 60°C. At the highest temperature, 90°C, with maximum addition of phosphoric acid, 2%, the produced lecithin color is completely black.

However, following disadvantages could be attributed to temperature of oil degumming (List, 1981; Shahidi, 2005):

- Susceptibility of oil to oxidation under higher temperatures.
- Higher level of free fatty acids in oil and lecithin.
- Decreasing of lecithin viscosity.

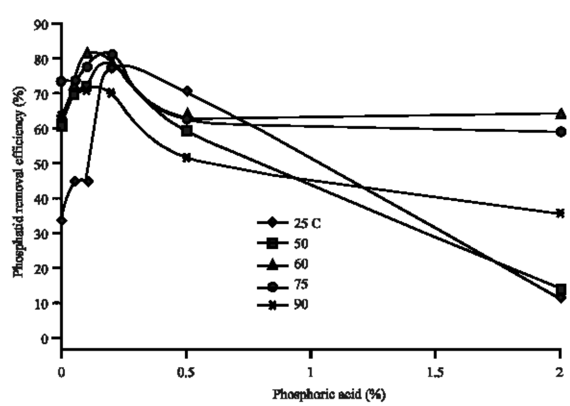

Fig. 3: Effect of increasing phosphoric acid percent on the phosphatid removal efficiency at different temperature

Table 5: Effect of water and phosphoric acid percents added to crude soybean oil on residual phosphatid at different temperatures

Temp	Run	Water (%)	Phosphoric acid (%)	Residual phosphatid (%)	Std. deviation	F	p-value	Sig
	1	3	0	0.9067	0.0305			
	2	2.95	0.05	0.7467	0.0776			
25	3	2.9	0.1	0.75	0.06			1,2 and 3 versus
	4	2.8	0.2	0.3067	0.075	103.482	p<0.001	4,5,6
	5	2.5	0.5	0.4067	0.0208			4 and 5 versus 6
	6	1	2	1.2067	0.0472			
	1	3	0	0.5367	0.04163			
	2	2.95	0.05	0.4167	0.04163			
50	3	2.9	0.1	0.3833	0.1002	60.814	p<0.001	1,5 versus 4,6
	4	2.8	0.2	0.3	0.01			2,3, 4 versus 6
	5	2.5	0.5	0.5567	0.1002			
	6	1	2	1.1733	0.0757			
	1	3	0	0.5007	0.01528			
	2	2.95	0.05	0.37	0.01732			
60	3	2.9	0.1	0.2533	0.03512	44.467	p<0.001	1,5,6 versus 2,3,4
	4	2.8	0.2	0.28	0.01			2 versus 3
	5	2.5	0.5	0.5	0.05568			
	6	1	2	0.49	0.02			
	1	3	0	0.3667	0.02082			
	2	2.95	0.05	0.36	0.05292			
75	3	2.9	0.1	0.31	0.02	54.164	p<0.001	1,2 versus 4,5,6
	4	2.8	0.2	0.2633	0.01528			3,4 versus 5, 6
	5	2.5	0.5	0.5167	0.01155			
	6	1	2	0.5633	0.02517			
	1	3	0	0.5067	0.01528			
	2	2.95	0.05	0.4067	0.02309			
90	3	2.9	0.1	0.3967	0.01528	171.805	p<0.001	1 versus 2,3,4,5,6
	4	2.8	0.2	0.41	0.04583			2,3,4 versus 5,6
	5	2.5	0.5	0.6633	0.01528			5 versus 6
	6	1	2	0.8833	0.02517			

Effect of adding phosphoric acid: The results presented in Table 5 and Fig. 3 show that by increasing phosphoric acid percent during lecithin production process, at first the efficiency increased strongly, but when phosphoric acid addition exceeds 0.2% an inverse result occurs i.e. phosphatid content of degummed oil increased. Phosphoric acid affects adversely the color of produced lecithin and turns it color severely darker. By adding 2% of it to the oil the color of produced lecithin is completely black. The conventional concentration of phosphoric acid used in oil degumming is 85 % and some authors propose lower acid concentration for higher lecithin quality (Bernardini, 1985; Shaaidi, 2005; Pan *et al.*, 2000).

CONCLUSION

In study of influencing factors on lecithin production it could be concluded that the lecithin production method by water is preferred to method of phosphoric acid. In fact, if the aim is producing oil with the minimum amount of residual phosphatid within it, addition of phosphoric acid will increase the efficiency of phospholipids separation by even elimination of non-hydrated phosphatids, but if the quality of separated phospholipids considered as lecithin needs to be take in account, addition of phosphoric acid wouldn't be recommended due to its effect on making the color of lecithin dark and presence of precipitated non-hydrated phosphatids in lecithin. However, the color of lecithin recovered from Soybean oil depends in many factors, which of the most important ones we can address to the quality of soybean utilized, age and origin of it. In this research by addition of 0.05% phosphoric acid or lesser amounts at 60°C and under stirring time of 20 min we obtained results similar to those by adding 3% water at 75°C and stirring time of 20 min. Addition of 0.1% and even 0.2% phosphoric acid reaches the efficiency of lecithin recovering to the higher levels, however, control of temperature to avoid lecithin discoloration to the dark color is extremely critical.

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