Iranian Journal of Chemical Engineering
Vol. 5, No. 1 (Winter), 2008, IAChE

Research note

Enhanced Degumming of Soyabean Oil and its Influences on Degummed Oil and Lecithin

*P. Eshratabadi[1], M. H. Sarrafzadeh[*2], H. Fatemi[2], M. Ghavami[3], N. Gholipour-Zanjani[1]*

1- Department of Food and Agriculture Institute of Standards and Industrial Research of Iran.
2- Department of Chemical Engineering, College of Engineering, University of Tehran, Tehran, Iran.
3- Science & Research Branch of Islamic Azad University,Tehran,Iran.

Abstract

In order to study the effective factors on the quality and quantity 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 a phosphorus content of 454 ppm was used for this purpose. Treatments were carried out under different concentrations of phosphoric acid (zero, 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) water at 75 °C with a stirring time of 20 minutes. Adding phosphoric acid decreased the phosphatid residue in the degummed oil, but the quality of lecithin was reduced. However, the addition of phosphoric acid lower than 0.05 %(v /v) at 60 °C resulted in the same recovery efficiency with no major effect on the quality of lecithin.

Keywords: *Soybean oil, Degumming, Lecithin, Phosphatid, Extraction*

Introduction

The contribution of food materials consumption in human health is an obviously accepted fact. Thus, incessant and extensive researches on improving food quality are always under consideration. Nowadays, the study of foods with natural origins have been allotted a special place of its own compared to that of artificial origin foodstuff, and valuable results are also obtained in this respect. Lecithin is an example of such materials as its natural origin has led to an increase in the popularity of those products that utilize it instead of synthetic compounds [1]. During the recent century humans have recognized the value of lecithin, and according to the research 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 lecithin quality and its yield extraction have always been a main fows of producers in various food industries [2-4].

The presence of phospholipids or phosphatids, or in other words, gums in vegetable oil cause a higher oil loss in the neutralization stage, and on the other hand,

∗ Corresponding author: E-mail: sarrafzdh@ut.ac.ir

generate numerous difficulties, while oil being transported from tanks and, even more, can obstruct the pipes completely. Therefore, by simple degumming not only would the above problems be prevented, but also the separated gum could be sold as commercial lecithin in the market [5-7]. The basis of extraction of phosphatids has relied on the principle that phospholipids become swollen when treated with hydrating substances, and are separated in the form of precipitation. For this, the hydrating material must be mixed with oil and then extracted after the sedimentation of phosphatids. Thus the insolubility of hydrated phosphatids in oil, and the higher specific gravity lead to a simple separation[8, 9].

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 the phosphatids of soybean oil, extracted from good quality seeds, are hydratable phosphatids. During appropriate water induced degumming, about 95-98% of phosphatids, which mainly consist of alpha lipoid, are separated [10]. The amount of added water for the hydration of phosphatids is very critical. If the water addition is lower than the needed amount, hydration will not happen appropriately, and if the amount of water is higher than its optimal value, high oil loss and a decrease in the percent of phosphatids in extracted gums results. In some oil extraction plants, the gums are hydrated using the condensed vapor of the misselate solvent extraction section. In this case the amount of added water should be controlled carefully due to direct vaporization, and it is often preferred that warm water be used instead[11]. 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[12]. 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 [11]. The aim of this paper 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 & Methods

Soybean crude oil with a phosphorus content of 454 ppm was used throughout this work. The phosphatid content normally could be calculated by multiplying the phosphorus content by 30 [13]. 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 analytical grade.

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

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

At first, for determining the optimal mixing time of soybean crude oil with hydrating material, experiments were carried out using water alone or diluted phosphoric acid as

hydrating agents. Other parameters were kept constant. Mixing time variations were 5, 10, 20, 40 and 60 minutes. After phase separation, the phosphorus content of degummed oil and the 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 minutes of stirring and with 3 repetitions for each.

Results and discussion

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

Effect of amount of water addition

As seen in figure 1, by increasing the amount of water added to crude oil the lecithin recovering efficiency increased and reached a maximum value of about 73 % at water percentage addition of 3. However, a higher increase in the amount of added water resulted in a lesser efficiency. In fact, if the water addition exceeds a certain limit, the water and oil emulsion could be formed and the efficiency of lecithin recovery would be decreased.

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

Regarding these results, it should be mentioned that despite some reports [3, 4] which suggest a water addition equal to the phosphatid content of the oil, the optimal value needs to be determined experimentally for any crude oil [11,14].

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}	450.982 P<001	0.5 and 1 versus all others. there is no significant differences between 2, 3 and 4
1	0.7167	2.517×10^{-2}		
2	0.4333	2.082×10^{-2}		
3	0.3667	1.032×10^{-2}		
4	0.4867	1.528×10^{-2}		

* one way analysis of variance

Influence of stirring time

By increasing mixing time and as a result of prolonging reaction time, it is logical to expect that the phosphatid removal efficiency will also be increased. However by considering the adverse effects of temperature on the color of lecithin over time, an optimal stirring time should be defined.

The results showed that there is a significant difference among the 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 systems, within the first 5 minutes, about 64% of gums are separated, and after 10 minutes and 20 minutes of stirring, the efficiencies increased to 69 and 73 % respectively, and maintaining stirring for one hour resulted in only about a 4 percent increase of phosphatid elimination. Statistical analysis (Table 3) showed that in this system there were significant differences between 5 and 10 minutes with 60 minutes, but there were no significant differences between 10, 20 and 40 minute stirring times ($P<0.05$). However, by considering the point that lecithin is a heat sensitive substance with dark discoloration, the stirring time of 60 minutes, in spite of generating a greater amount of phosphorus elimination, is not suitable for lecithin recovery.

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

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 significant differences between 3,4 and 5.
3	0	4	40	0.3500	3×10^{-2}		
3	0	5	60	0.3100	2×10^{-2}		
2.9	0.1	1	5	0.2933	1.528×10^{-2}	8.566	1,2 and 3 versus 4 and 5.
2.9	0.1	2	10	0.26	0.01	P<0.003	There is no significant difference between 4 and 5.
2.9	0.1	3	20	0.25	3×10^{-2}		
2.9	0.1	4	40	0.24	3.6×10^{-2}		
2.9	0.1	5	60	0.16	5.19×10^{-2}		

* Multiple comparisons, dependent variable: phosphatid residue, scheffe.

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 *
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 X 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

Effect of Temperature

By increasing temperature, the percent of phosphatids elimination from soyabean crude oil should be increased. Our results showed that in a water system at 25 °C, the efficiency of lecithin production is only 33%, and by increasing the 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 deeply dark. When, with increasing temperature the phosphoric 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%, 80.9%, respectively).

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

However, the following disadvantages could be attributed to the temperature of oil degumming [10,11]:

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

Effect of adding phosphoric acid

The results presented in Table 5 and figure 1 show that by increasing phosphoric acid percent during the 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 adversely affects the color of produced lecithin and turns its color severely darker. By adding 2% of it to the oil, the color of the produced lecithin is completely black. The conventional concentration of phosphoric acid used in oil degumming is 85 % and some authors propose a lower acid concentration for higher lecithin quality [5,11,15].

Conclusion

In this study of influencing factors on lecithin production it could be concluded that the lecithin production method by water is

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

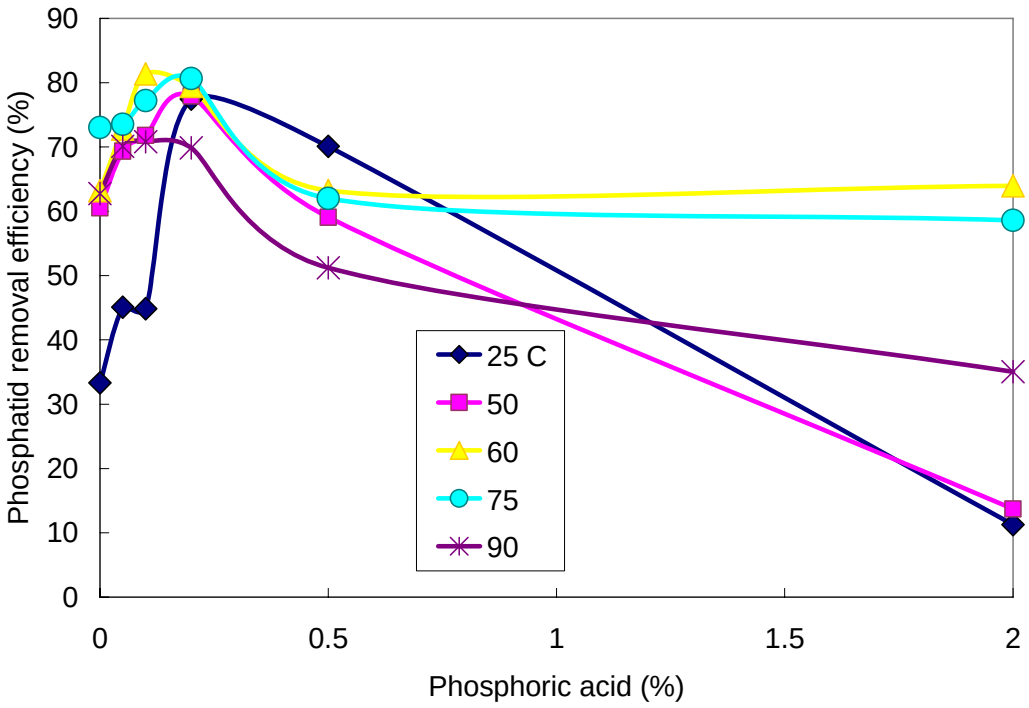

Figure 1. Effect of increasing phosphoric acid percent on the phosphatid removal efficiency at different temperature.

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

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

* Analysis of variance – Multiple comparisons

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

References

1. Wendel, A., "Lecithin: The first 150 years," INFORM, 11(8), 885 (2000).
2. Wu, Y., and Wang, T., "Soybean lecithin fractionation and functionality," JAOCS, 80(4), 319 (2003).
3. Arvanitoyannis, I.S., "Emulsifiers in food technology," International Journal of Food Science and Technology. 40 (4), 464(2003).
4. Orthoefer, F., and List, G. R., "phospholipids/ Lecithin: A class of nutraceutical lipids," Boca Raton, CRC press, p.509 (2006).
5. Bernardini, E., "Vegetable oils and fats processing," 1th ed., Interstampa, Rome, Italy, p104 (1885).
6. Krawezyk, T., "Lecithin: Consider the possibilities," INFORM.,7(11)., p. 1158 (1996).
7. Wendel, R., "Lecithin,in Kirk-Othmer Encyclopedia of chemical technology," 14th ed., John Wiley & Sons, New York, p. 250 (1984).
8. Wiedermann, L.H., "Degumming, Refining and Bleaching Soybean oil," JAOCS., 58(3).,159 (1981).
9. Haraldsson, G., "Degumming, dewaxing and refining," JAOCS.,60(2).,251(1983)
10. List, G.R., "Effect of degumming conditions on removal &quality of soybean lecithin," JAOCS, 58(10)., 892(1981).
11. Shahidi, F., "Baileys'industrial oil and fat products," 6th ed. John Wiley & Sons, Newfoundland, Canada, V2, p.604, (2005).
12. Smiles, A., Kakuda, Y., Macdonald, B.E., "Effect of degumming reagents on the composition and emulsifying properties of canola, soybean and sunflower acetone insolubles," JAOCS., 66(3), 348 (1989).
13. Racicot, L.D., Handal, A.P., "Degumming of soybean oil: quantitative analysis of phospholipids in crude and degummed oil," JAOCS., 60(6).,1098 (1983).
14. Indira, T.N., Hemavathy, J., Gopala, A.G., Bhattacharya, S., "Water degumming of rice bran oil: A response surface approach," Journal of Food Engineering., 43(2)., 83 (2000).
15. Pan, L.G., Canpana, A., Toms, M.C., M.C.A.N., "Kinetic study of phospholipid extraction by degumming process in sunflower seed oil," JAOCS., 77(12).,1273 (2000).