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Process Conditions for Maximum Yield of Crude Soy Lecithins and Its Quality Improvement by Solvent Extraction

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조 레시틴의 제조공정 조건과 용매추출에 의한 품질향상

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Abstract

A study was conducted for process conditions for maximum yield of crude soy lecithins and its quality improvement by solvent extraction. Results indicate that maximum yield of the crude lecithins was obtained by hydration with 3% water at degumming temperature 75°C. On the other hand, water level used for hydration was found to affect the recovery yield of the lecithins more profoundly than the degumming temperature. It was also found that extractions with acetone and 2-propanol increased the phosphatidyl choline content from 19.7% to 28.3% and 58%, respectively.

Introduction

Currently potential production of soy lecithins is estimated to be at least 1,000 tons per year in Korea based on the annual soybean crushing amount by soybean industry[1]. Although there is a demand for food- and pharmaceutical-grade lecithins, the crude lecithins, a by-product from soy oil degumming process, is utilized mainly as an ingredient of feed and to a much less extent, as a food emulsifier, since their quality is not up to par. Instead, quite a substantial amount of the food-grade lecithins are imported from other countries. Therefore, it is imperative to upgrade the quality of the locally produced soy lecithins to meet the requirement not only as food ingredients but also as the ingredient of pharmaceutical and other applications[2~6]. By upgrading their quality, we can accomplish two objectives. First objective is to maximize the utilization of the expensive soy beans imported from other countries, thus resulting in saving, and second objective is to supply possibly less expensive lecithins of a better quality to food and pharmaceutical

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industries.

In the assessment of quality of soy lecithins, the color is identified as the most troublesome and the process conditions such as degumming temperature and vaccum drying time and temperature are reported to affect the color most profoundly mainly due to the browning reaction[7~10].

Another aspect of the soy lecithin quality is related to its chemical composition[11]. Commercially available crude soy lecithins are mixture of phosphatidyl choline, phosphatidyl inositol and other minor phosphatides with 35% neutral oil. This crude soy lecithins are further processed to nearly oil free lecithins(3~4% neutral oil) by acetone extraction and this product favors either o/w or w/o emulsions. Then an alcohol extraction step would separate the oil-free lecithins into an alcohol-soluble fraction, enriched in phosphatidyl choline and the alcohol-insoluble fraction, enriched in phosphatidyl inositol and phosphatidyl ethanolamine. The former would favor o/w emulsion and the latter would favor w/o emulsion. As indicated above, the soy lecithins of various types are commercially produced in such advanced countries as USA, Japan and European countries. However, detailed conditions of the soy lecithin process for the maximum yield, reduction of the color, and refining for better emulsifying properties of soy lecithins are not available in the literature. Therefore, we conducted a series of tests to identify process variables for the maximum production and better quality in terms of color and emulsifying properties.

Meterials and Methods

Materials

Crude soy oil obtained from soy oil refinery of Cheil Sugar Co. at Inchon was used as a starting material. Phosphatidyl choline, phosphatidyl ethanolamine, phosphatidyl inositol, triolein, sphingomyelin, cerebrosides, phosphatidyl serine and cardiolipin were the products of Sigma (St. Louis, MO, USA). The solvents for HPLC were chromatographic grade and were the products of either Merck (Darmstadt, W. Germany) or Burdick & Jackson (Muskegon, MI, USA). A RC-5 superspeed refrigerated centrifuge(Sorvall) and a vacuum dryer (Forma Scientific Model 6414, Marietta, OH, USA) were used for separation of wet gums from the hydrated crude oil and for drying the wet gums, respectively.

Separation of crude soy lecithins from crude oil

500 *g* of the crude oil was mixed thoroughly in a 1 *l* beaker with 1~8% water with 1% increment at temperature from 35 to 85°C with 10°C increment for 30 min. The stirring speed was 100 rpm. The phosphat ides were hydrated to form a gummy sludge and the hydrated sludge was removed from the oil by centrifugation at a speed of 500 rpm for 20 min. Then the lecithin sludge containing 40~50 % water was dried to 1% moisture content by vacuum drying. Moisture, acetone-insoluble matter, total phosphorus and color of the dried soy lecithins were analyzed by the methods of AOCS[12].

Solvent extraction of crude soy lecithins

Crude soy lecithins (dried gums) obtained were extracted with acetone to remove neutral oil by the method of Weenink *et al*[13]. The acetone-insoluble fraction, in turn, was extracted with 2-propanol by the method of Liebing[14].

Quantitative analysis of phosphatidyl choline by HPLC

Phosphatidyl choline content for crude soy lecithins, acetone-insoluble fraction, and 2-propanol-soluble fraction were determined by HPLC (Waters Associates, ALC/GPC-244, Milford, MA, USA) packed with μ-porasil column(Waters Associates). The operating conditions were: flow rate, 2 *ml*/min; solvent, chloroform : methanol : acetate : water(14 : 14 : 1 : 1, v/v/v/v); detector, RI 104; attenuation, 32×.

Results and Discussion

Soy lecithin processing

(1) Effects of water level used for hydration and degumming temperature on the yield of soy lecithins

Fig. 1 shows acetone-insoluble fraction of dried

gums recovered (%) and recovery yield of phosphatides (%) as a function of water levels used for hydration (%) at degumming temperature 75°C.

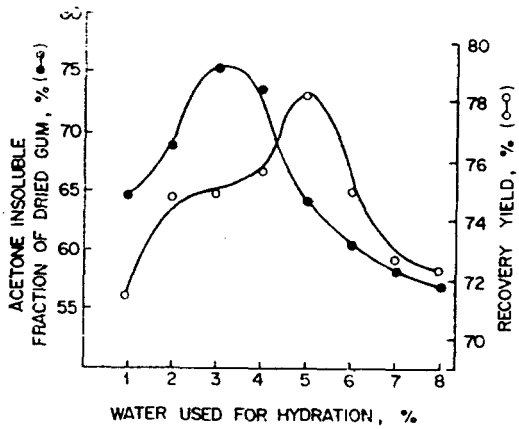

Fig. 1. Acetone-insoluble fraction of dried gums recovered and recovery yield of phosphatides as a function of water level used for hydration at degumming temperature 75°C

The results indicate that the maximum acetone-insoluble fraction of dried gums was 75% at 3% water level and only 64% acetone-insoluble fraction of dried gums was obtained at 5% water level. However, maximum recovery yield of phosphatides was 78% at 5% water level, and the recovery yield of phosphatides was 74.5% at 3% water level. Therefore, it can be concluded that the hydration with 3% water is optimal for recovery of the phosphatides.

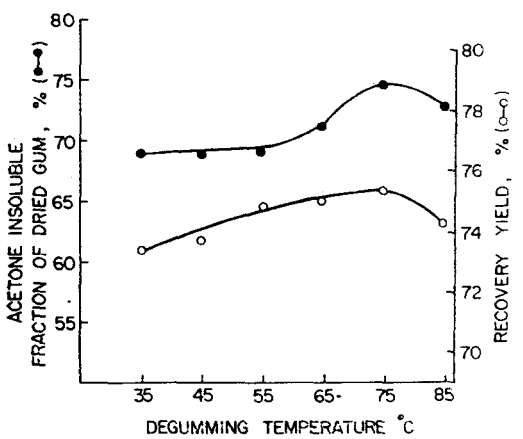

Fig. 2. Acetone-insoluble fraction of dried gums recovered and recovery yield of phosphatides as a function of degumming temperature with 3% water for hydration

Fig. 2 shows acetone-insoluble fraction of dried gums and recovery yield of phosphatides as a function of degumming temperature with 3% water for hydration. As shown in Fig. 2, the degumming temperature did not influence significantly both acetone-insoluble fraction of dried gums and recovery yield of phosphatides, although the acetone-insoluble fraction of dried gums and the recovery yield of phosphatides were maxima at 75°C. Therefore, degumming temperature was not as important as water level for hydration for the recovery yield of phosphatides. However, it was found that the low degumming temperature caused the separation of the gummy material from the degummed crude oil very difficult because of the increased viscosity and a degumming temperature of 75°C appears to be optimal for the separation of wet gums from the crude oil.

(2) Effect of degumming temperature, and vacuum drying temperature and time on color of soy lecithins

Fig. 3 shows the relationship between the color of wet gums and degumming temperature with 3% water for hydration. The result indicates that the

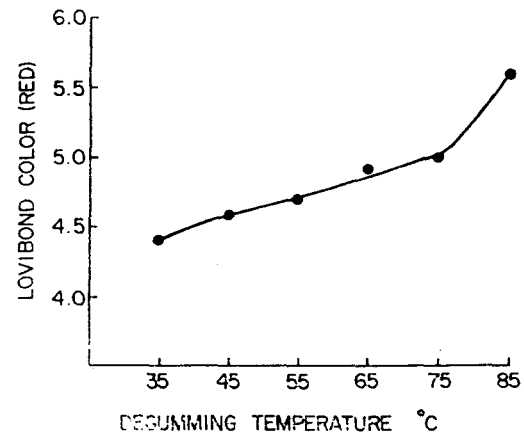

Fig. 3. Relationship between color of wet gum and degumming temperature with 3% water for hydration

Lovibond color of wet gum solution(1g/100 *ml* of mixed solvent(CH_3COOH $CHCl_3$=3 : 2)) was measured in 5 1/4 inch cell (Lovibond color matched against 15Y slide).

color of wet gums increased linearly as the temperature increased up to 75°C and the color increased at a higher rate at the temperature above 75°C.

Fig. 4 shows the relationship between the color of the dried gums and vacuum drying temperature with vacuum of 29 inches Hg vac. for 2 hr and Fig. 5 shows the relationship between the color of the dried gums and vacuum drying time with vacuum of 29 inches Hg vac. at 75°C. Both figures reveal that the color of the dried gums was increased linearly with the increase of both vacuum drying temperature and time. However, effect of the vacuum drying time on the color was much more pronounced than that of the vacuum drying temperature. This proves that the wet gums from the degumming process should be dried in vacuo at a high temperature for as short a time as possible within a practical vacuum range.

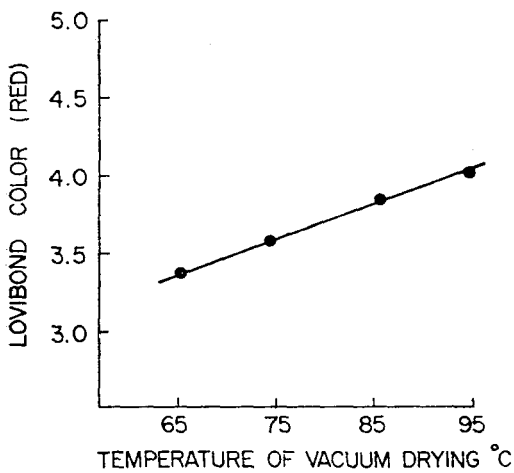

Fig. 4. Relationship between color of the dried gums and vacuum drying temperature with the vacuum of 29 inch Hg for 2hr
Lovibond color of dried gum solution (1 *g*/100 *ml n*-hexane) was measured in 5 1/4 inch cell(Lovibond red color matched against 15Y slide).

Analysis of phosphatidyl choline for crude lecithins, acetone-insoluble fraction and 2-propanol-soluble fraction.

In order to concentrate phosphatidyl choline in the crude soy lecithins, two fractions were separated from the crude soy lecithins. One fraction was obtained by acetone extraction of the crude soy lecithin followed by evaporation of the solvent. The other fraction was obtained by 2-propanol extraction of the acetone-insoluble fraction followed by evaporation of the solvent. Table 1 shows phosphatidyl choline content for each of the three samples. The results indicate that crude soy lecithins, acetone-insoluble fraction (oil-free lecithin), and 2-propanol-soluble fraction contained 19.7, 28.3, and 58.0%, respectively. Thus, 2-propanol-

Table 1. Phosphatidyl choline content* determined by HPLC of crude soy lecithins and lecithins extracted by solvents

Sample	Concentration (%)
Crude lecithins	19.7
Acetone-insoluble fraction	28.3
2-propanol-soluble fraction	58.0

$$\text{* Content}(\%) = \frac{\text{Phosphatidyl choline}(g)}{\text{Lecithins }(g)} \times 100$$

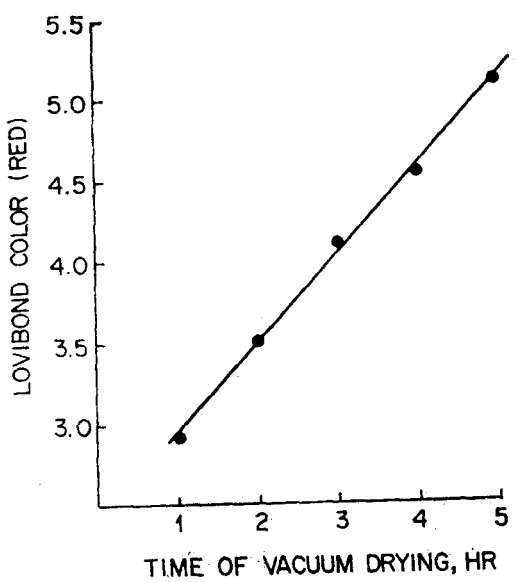

Fig. 5. Relationship between color of dried gums and vacuum drying time with vacuum of 29 inch Hg at 75°C
Lovibond color of dried gum solution (1 *g*/100 *ml n*-hexane) was measured in 5 1/4 inch cell(Lovibond red color matched against 15Y slide).

soluble fraction contained phosphatidyl choline three times higher than the crude lecithins.

요 약

대두레시틴의 제조공정 조건 규명과 용매추출에 의한 그의 품질향상을 꾀하기 위하여 실험실 규모로 실험을 행하였다. 실험결과 75°C에서 3%의 물로 수화하여 탈검 하였을 때 가장 많은 양의 레시틴을 얻었으며 대두레시틴의 조수에는 탈검 온도보다 물의 양이 더욱 큰 영향을 미치는 것으로 밝혀졌다.

유화특성을 개선하기 위하여 아세톤 추출과 이소-프로필 알콜 추출을 하였는바 이때 도스파티딜 콜린의 양은 19.7%에서 아세톤불용물의 경우 28.3%로 증가했고 이소 프로필 알콜 가용물의 경우 58%까지 증가함을 알 수 있었다.

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