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Dewaxing of Sunflower Seed Oil

Joon-Shick Rhee
Department of Biological Science & Engineering
Korea Advanced Institute of Science, Seoul
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해바라기 油의 탈납

이 준 식
한국과학원 생물공학과
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Abstract

By using the existing caustic refining system with a minimum modification and by using a combination of various emulsifiers(0.2 % sodium hexametaphosphate, 0.05 % sodium lauryl sulfate and 0.001 % dioctyl sodium sulfosuccinate), a new economical dewaxing process for sunflower seed oil was developed in order to reduce the cost of the dewaxing process. The results indicate that the waxes can be removed satisfactorily from the sunflower seed oil by emulsifying, batchwise or continuously, the oil with the aqueous surfactant solution, followed by centrifugation at ambient temperatures (16~27°C).

Dewaxing loss for the batch process was satisfactory for both low wax- and high wax-crude oil, whereas dewaxing loss for the continuous process needs to be improved. The results indicate that initial level of wax content (low wax vs. high wax) did not affect the loss for batch process (0.82 % vs. 0.62 %), but affected the loss for continuous processes, regardless of the type of mixing mode (2.28 % and 5.68 % for low wax-and high wax-oil, respectively).

It was also noted that the type of mixing mode (centactor vs. static mixer) for the continuous process affected the loss, regardless of the wax content (5.2 % and 2.8 % for contactor and static mixer, respectively).

Introduction

Edible vegetable oil such as sunflower seed oil, rice bran oil, safflower oil and corn oil contain a significant fraction of waxes (2-5 %) originated from the hull, pericarp of the kernel and kernel itself in the course of pressing and/or solvent extraction.[1] The waxes from the edible vegetable oils (sunflower seed oil and rice bran oil) are similar in characteristics to the commercially important waxes (Table 1)[2,3].

These waxes are very difficult to be removed by the conventional oil refining processes such as caustic refining, clay bleaching and deodorization and cause the oil to become cloudy, when stored at refrigeration temperature or even at room temperature, thus resulting in the negative consumer responses.

Consequently, waxes had to be removed by the additional dewaxing process; winterization followed by filtration at low temperature(0-4°C)[4]. However, this process is costly not only due to the refrigeration required and limited throughput, but also due

Table 1. Characteristics of waxes from sunflower and rice bran oils and of commercially important waxes [2,3]

Characteristics	Sunflower	Rice bran	Bees	Carnauba	Candelilla	Ouricury
Specific gravity	0.97	—	0.96	0.99	0.97	1.00
Meltine point(°C)	73 ~81	75 ~80	63 ~ 64	82 ~84	68 ~73	79 ~80
Acid value	9.5~26	4 ~14	20 ~ 23	1 ~ 8	14 ~19	15 ~20
Saponification value	88 ~90	77 ~85	96 ~107	78 ~86	54 ~67	66 ~72
Iodine value	8 ~12	11 ~16	7 ~ 10	9 ~13	14 ~20	6 ~ 7
Unsaponifiables(%)	52 ~53	57 ~67	50 ~ 52	53 ~55	65 ~74	28 ~34

to the greater losses and extra filtration difficulties.

In order to minimize the processing cost, a new dewaxing process was developed by using the existing caustic refining system with a minimum modification and by using a series of emulsifiers (sodiumlauryl sulfate, sodium hexametphosphate and diocty lsodium sulfosuccinate)[5,6]. The purpose of this paper is to present the results of the new process development.

Experimental

1. Materials

Six 55 gallon drums of each low wax- and high wax-crude sunflower seed oil were charged to two different 700 gallon holding tanks, respectively. After the oil from each tank was thoroughly mixed by a recycle pump for approximately 10 hours, representative samples were taken from each tank to be analyzed for wax content[5], free fatty acid[7a], moisture[7b], phosphorous[7c], peroxide value[7d], specific gravity[7e], viscosity[7f], iodine value[7g], and fatty acid composition.[7h] Analytical results shown in Table 2 indicate that the two samples were of the same quality, except the wax levels. Various surfactants were tried in bench scale and the combination of sodium hexametaphosphate, sodium lauryl sulfate,

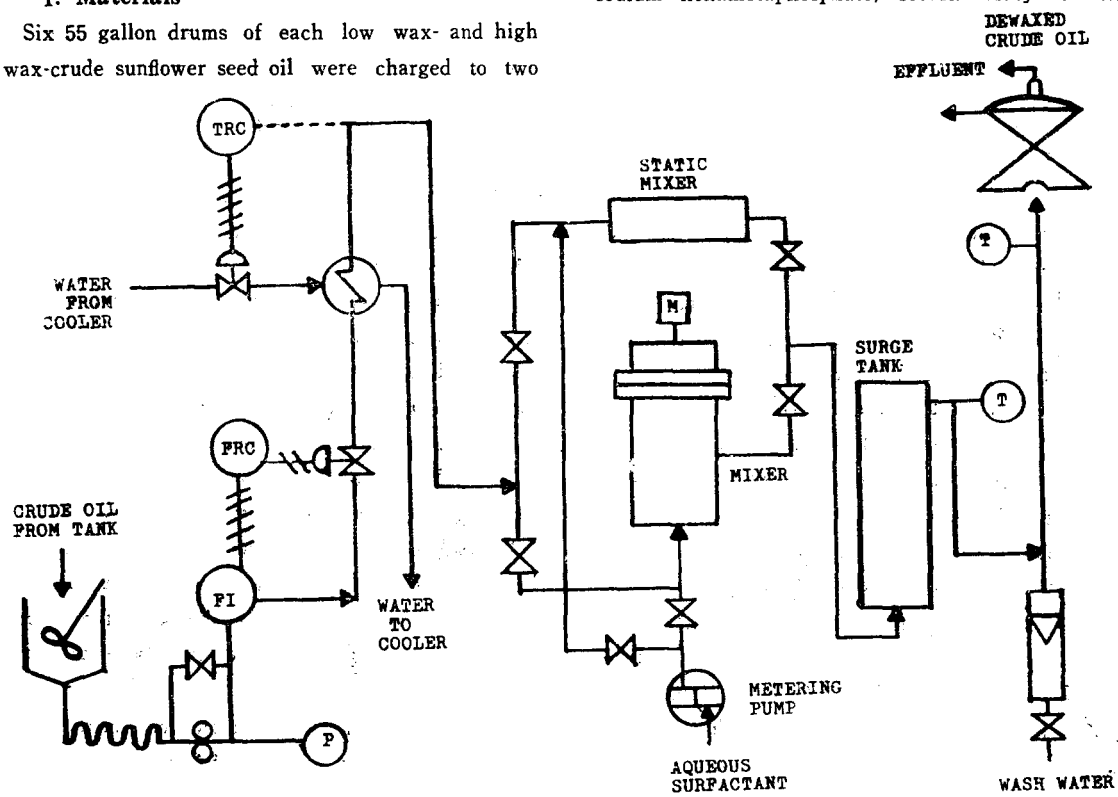

Fig. 1. Schematic sunflower oil dewaxing process

and dioctyl sodium sulfosuccinate was found to be most effective.

2. Equipment

The dewaxing system was essentially the same as pilot scale 180 kg/hr. continuous caustic refining system with a minimum modification(see Figure 1). A cooler (Whitlock AHT-1-A-SS, 6-W-36) was used in order to maintain the oil temperature below 25°C. A custom built contactor or a static mixer (3/4″ pipe with 6 elements manufactured by Koch Engineering Co.) was used to emulsify crude oil with aqueous surfactant solution. A metering pump (Lapp's pulsa-feeder model CP-1) was used to feed the aqueous surfactant solution. Westfalia's self cleaning centrifuge (model SAOOH-205) was used for separation of dewaxed crude oil from aqueous waxy effluents.

Table 2. Analysis of low wax- and high wax-sunflower oils

	low wax	high wax
Wax contents(%)	0.15	1.95
Free fatty acids(%)	0.69	0.72
Moisture(%)	0.08	0.07
Phosphorous(ppm)	200	226
Peroxide value(meq/kg)	10.9	12.9
Specific gravity @ 24°C	0.9214	0.9218
Viscosity @ 24°C (cp)	44	46
Iodine value	138.0	138.9
Fatty acid composition(%)		
Palmitic 16:0	6.1	6.2
Stearic 18:0	4.6	4.2
Oleic 18:1	17.5	17.0
Linoleic 18:2	71.0	71.0

Table 3. Process conditions for sunflower dewaxing

Process type	Batch		Continuous			
Wax content of crude oil	Low	High	Low		High	
Mixing mode	Agitator	Agitator	Contactor	Static	Contactor	Static mixer
Flow rate of crude oil & surfactant mixture (kg/hr)	114.0	114.5	114.5	114.5	114.5	114.5
Flow rate of city water (kg/hr)	22.7	22.7	22.7	22.7	22.7	22.7
Centrifuge temperature (°C)	16~27	18~26	18~24	20~26	16~23	17~24
Line pressure (atm)	3.1~3.8	3.1~3.8	3.1~3.8	3.1~3.8	3.1~3.8	3.1~3.8

3. Procedures

A series of dewaxing tests was conducted for each low wax- and high wax-sunflower oil samples (See Table 3 and Fig. 2 for process conditions). The important process varipbles are mixing mode, flow rate of crude oil and aqueous surfactant solution, flow rate of water to lower centrifuge temperature, centrifuge temperature and line pressure.

Three different mixing modes were tried to evaluate the degree of dewaxing and the dewaxing loss; (1) 95 % crude oil was mixed with 5 % aqueous surfactant solution by a slow rotating agitator and was held for 30 minutes (maintaining at 24°C), followed by centrifugation, (2) 95 % crude oil (maintaining @ 24°C maximum) and 5 % aqueous surfactant solution were mixed in a contactor continuously and the mixture was held in a surge tank for 6 minutes, followed by centrifugation, (3) the same process as (2), except using a static mixer instead of a contactor for mixing crude oil and aqueous surfactant solution. Aqueous surfactant solution consisted of 94.98 % water, 4.00 % sodium hexametaphosphate, 1.00 % sodium lauryl sulfate and 0.02 % dioctyl sodium sulfosuccinate. Approximately 20 % city water was added to the mixture of 95 % crude oil and 5 % aqueous surfactant solution just before centrifugation to lower the temperature at which the dewaxed oil was separated from aqueous waxy effluents.

All dewaxed crude oils were caustic-refined by using the same condition before wax determination since the dewaxed oils out of the centrifuge were turbid because of the moisture and emulsifier residues.

The caustic refining was carried out in a 3 liter separatory funnel by using the following condition:

Caustic strength ('Be) 16

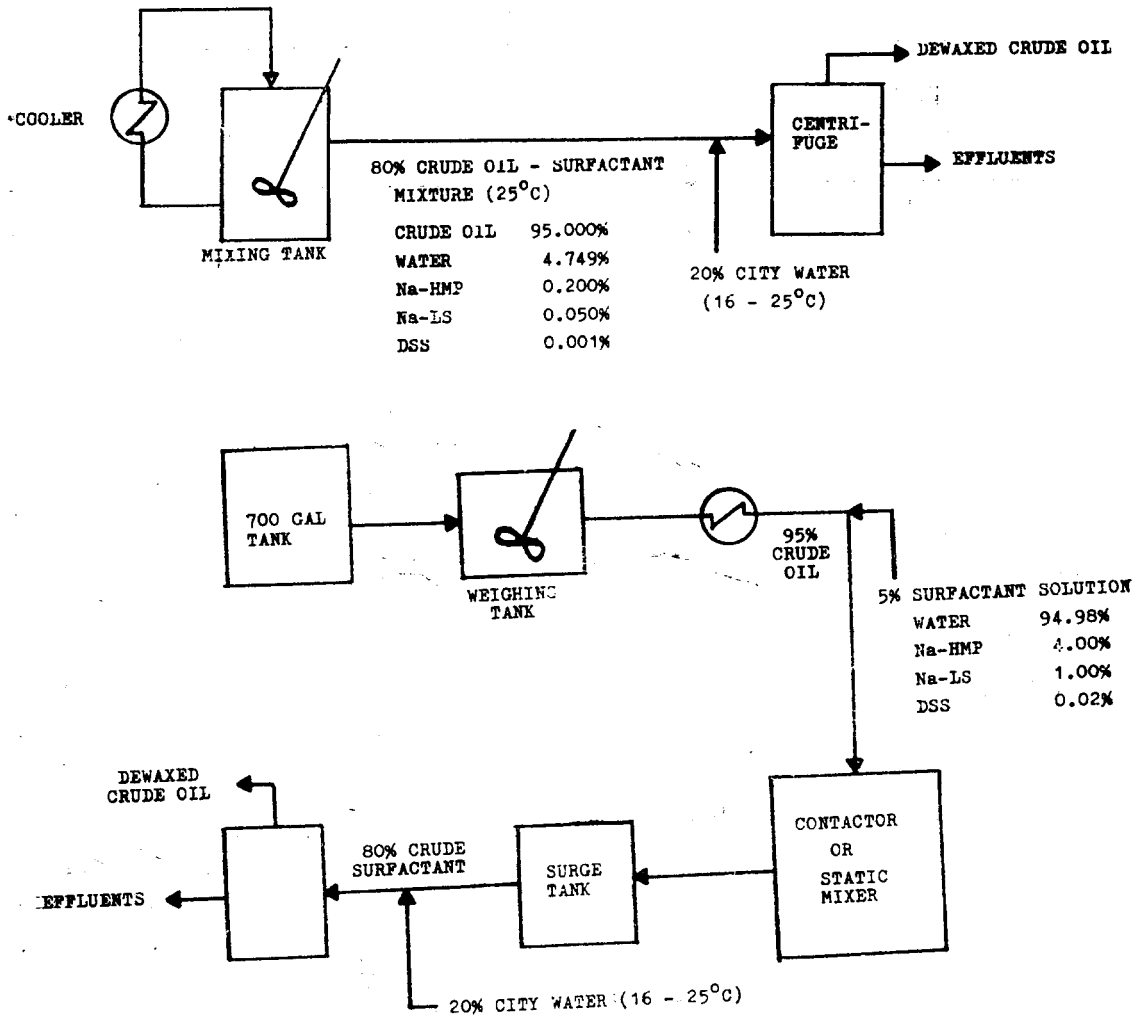

Fig. 2. Schematic flow diagram for sunflower oil dewaxing processes

Excess caustic (%)	0.12
Refining temperature (°C)	85
Number of water washing	12
Temperature of dehydrating & filtration @ 28"Hg Vac. (°C)	85

Results & Discussion

Wax contents of the dewaxed and caustic-refined oil samples were qualitatively determined by two different methods; cold hexane extraction followed by gravimetric determination[5], and periodical visual examination of the oil placed in the ice chest[7i].

At present time, there is no quantiative method of wax determination for such a low level of wax content as in the dewaxed oil. Our experience has shown that the oil was visually free of waxes, when the wax content by the cold hexane extraction followed by gravimetric determination was less than 60ppm. Table 4 shows that all of the dewaxed and caustic refined oils were visually free of waxes. All dewaxed and caustic refined oils were brilliant and clear when placed in ice chest for four days. Undewaxed controls for low wax- and high wax- oils (caustic-refined

Table 4. Wax contents of dewaxed sunflower oil and dewaxing loss

Process type	Batch		Continuous			
Wax contents of crude oil	Low	High	Low		High	
Mixing mode	Agitator	Agitator	Contactor	Static mixer	Contactor	Static mixer
Wax contents of dewaxed crude oil gravimetric (ppm)	50	29	1	1	39	3
Dewaxing loss (%)	0.82	0.62	2.73	1.83	7.67	3.68

only) were very hazy, even before being placed in the ice chest.

Throughout the test, flow rates of crude oil and aqueous surfactant solution and city water were fixed at constant value (see Table 3). The flow rate of dewaxed crude oil and aqueous waxy effluents were monitored in order to obtain the dewaxing loss. Table 4 shows the dewaxing loss for each of the test series. It was found that the oil loss in the batch process was substantially lower than the losses in continuous proces, especially in the case of high wax content oil. It appears that a certain elapsed time was required between mixing the oil and surfactant solution and centrfugation to allow wax molecules to transfer to the interfaces between oil and water phases and to allow the emulsion loose, thus resulting in the better separation of the oil phase from the aqueous waxy phase. The elapsed time between the mixing and centrifugation for batch process was about 30 minutes, whereas the elapsed time for continuous processes (using a contactor or a static mixer) was about 6 minutes.

It was also noted that the oil loss from the static mixer was lower than that from the contactor for the continuous process. The reason for the lower loss from the static mixer than from the contactor appears to be due to the fact that the tighter emulsion was formed when the contactor was used and consequently, it was difficult to separate the aqueous waxy phase from the oil. This was especially true with high wax crude oil.

Thus, waxes can be removed satisfactorily from the crude sunflower oil by emulsifying, batchwise and continuously, the oil with surfactant solution, followed by centrifugation at ambient temperature (16~27°C).

Dewaxing loss for the batch process was satisfactory for both low wax- and high wax-crude oils, whereas dewaxing loss for the continuous process needs to be improved. The results indicate that initial level of wax content (low wax vs. high wax) did not affect the dewaxing loss for batch process (0.82 % vs. 0.62 %), but affected the loss for continuous processes, regardless of the mixing mode(2.28 % and 5.68 % for low wax- and high wax- oils, respectively). It was also noted that the type of mixing mode (contactor vs. static mixer) affected the dewaxing loss, regardless of the wax content: the static mixter was more efficient in reducing the loss than the contactor (5.2 % vs. 2.8 %). To reduce the loss for continuous process to the level comparable to that for batch process (below 1 %) and yet to dewax satisfactorily, further study is required in the following areas:

(1) Waxes are to be settled as much as possible and separated from the crude oil and then proceed the dewaxing process as described.

(2) It was evident that the static mixer was better in reducing the loss than the contactor. In this test, six elements of the static mixter were used and it appears that the less number of the elements should be used to reduce the oil loss to the level comparable to that for batch process.

(3) A larger insulated surge tank is installed to increase the elapsed time between mixing and centrifugation to the level comparable to that for batch process (i.e. 30 minutes).

In conclusion, waxes can be removed satisfactorily from the crude sunflower seed oil by the new process described in this paper which would be more efficient and more economical in comparison to the conventionall dewaxing process.

요 약

해바라기유의 재래식 탈납공정의 경제성을 높이기 위해서 여러가지 유화제(0.2% sodium hexametaphosphate, 0.05% sodium lauryl sulfate, 0.001% dioctyl sodium sulfosuccinate)를 이용한 새로운 탈납공정을 개발하였다.

해바라기 원유를 회분식(batch process)과 연속식(continuous process)으로 유화제 수용액과 유화시킨후 원심분리기로 분리시켰을 때 재래식 탈납공정에 비견할 만한 탈납을 할 수 있음을 알았다. 탈납 공정에서 위에 설명한 탈납정도(degree of dewaxing)외에 또 하나의 중요인자는 수율(dewaxing loss)인바 회분식 공정의 경우에는 재래식 탈납공정의 수율과 같았으나 연속식 공정의 경우에는 개량할 여지가 있음을 알았다. 실험결과에 의하면 회분식 공정의 경우 탈납전 원유에 함유된 납(wax)의 다과에 관계없이 수율은 0.62~0.82%로 재래식 공정과 같았으나 연속식 공정인 경우에는 탈납전 원유에 함유된 납의 양이 적은 경우와 많은 경우 각각 2.28%와 5.68%로 현격한 차이를 보였다.

또한 연속식 공정의 경우 원유와 유화제를 유화시키는 방법에 따라 수율에 많은 영향이 미치는 것으로 밝혀졌으며 contactor와 static mixer를 썼을때 각각 5.2%와 2.8%이었다.

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   (d) Method Cd 8-53
   (e) Method Cc 10a-25
   (f) Method Tq 1a-64
   (g) Method Cd 1-25
   (h) Method Ce 1-62
   (i) Method Cc 11-53