METHOD OF ADDING OF THE POWDER CATALYST IN THE PROCESS OF THE HYDROGENATION OF FATTY ACIDS AND TECHNICAL GRADE FATS

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Economical utilization of fatty raw materials aims at the increase of the supply of edible fats and the outmost application of all grades of waste fats for the technical purposes [2].

Waste fats before application in the numerous technical fields must be subjected to the avrious refining processes to get their concistency teristics. One of the important property of fats is their consistency [1, 5].

Paper refers to the manufacturing of fats used as the addition to the soap feed-stock and also to the production of the stearin type fatty acids with the iodine value of 0-10. Both products were obtained in the hardening process. Technical grade fats or fatty acids obtained from them by splitting and distillation were used as raw materials in hardening process [6].

Hardening process is usually preceded by the refining, by which inorganic compounds, protein and mucilaginous materials and also water are removed. Fatty acids, after splitting process, are furthermore purified by distillation.

Despite of the application prior hardening the multi-stage purification of the technical grade fats or fatty acids not always is possible to maintain the standard quality of the particular raw material. In case of the application of the continuous processes standard quality of raw materials is an essential property.

Though the continuous processes are more economical at the processing of 15,00 tons (year of raw material, the situation resulted has made us to consider the application of the batch system as an equivalent solution of the problem [3].

Method presented hereby applied in the batch process gives as well

the better economics as the technological effects consisted in obtaining products with low idoine value, even for raw materials which are hard to hydrogenate.

It is known that in order to get products with low iodine value from low grade raw materials, it is necessary to use the catalysts of high activity. These catalysts however, are easily disactivated during the process. Method presented hereby is based upon retaining the high activity of the catalyst during its contact with the raw material [4].

Improvement of the typical basic technological process is based upon application of the two-stage hardening of the refined technical grade fats or distilled fatty acids. The first stage is the "preliminary hardening" and the second one the "final hardening". The first stage is carried out in the presence of the catalyst which has been already used in the identical process. This catalyst is obtained from the foregoing lot of the hardened fat or fatty acid. This stage is carried out for 0.5-1.5 hour at 150-180°C under the hydrogen pressure 2-5 atmospheres for fats and of about 20 atmospheres for fatty acids. Under such conditions of the reaction fat is refined from dissolved substances which are toxic to the catalyst. It makes possible to get better resuls in the second stage of the hardening than it is possible under known until now conditions of the single-stage hydrogenatoin. The second stage of the hardening of fats or fatty acids is carried out after introduction of the fresh catalyst in the amount equivalent to the 0.05-0.10% of nickel. Catalyst is distributed in fat or in fatty acid which has already had contact with the catalyst. This stage is carried out for 3-6 hours at the temeprature up to 200°C under the same pressure of hydrogen as during the first stage.

Experimental data presented hereby show the results of the hardening of technical grade lard, carried out under the largelaboratory and industrial scale conditions, and also the results of the large-laboratory scale hardening of fatty acids. Medium and high active catalysts and electrolytic hydrogen have been used in each case.

Results of four runs of hardening of the technical grade lard carried out under laboratory scale conditions in the 10 l autoclave and also under the industrial scale conditions for the charge of 7.3 tons are presented in Table 1. The catalyst obtained from the formerly made lots has been used in the first stage of the "preliminary hardening" carried out at 150-170°C.

Laboratory process was carried out under the standard conditions for 1 hour. Indutrial scale process was carried out for minimum 0.5 h up to 1.5 h. Slight increase of the temperature was observed. In the second stage of the hardening carried out under the laboratory scale conditions,

Table 1

Hardening of the technical grade lard

Description	Large-laboratory scale process				Industrial scale process			
	I	II	III	IV	I	II	III	IV
Parameters of the process								
1st stage: % of used Ni	0.06	0.10	0.10	0.10	about 0.05	about 0.08	about 0.05	about 0.08
Temperature, °C	150—180	150—180	150—180	150—180	150—180	150—180	150—180	150—180
Pressure, atm.	3	2	2	2	about 1.5	about 1.5	about 1.5	about 2.5
Time, h	1	1	1	1	1	1	1	1
2nd stage: % of fresh Ni	0.05	0.10	0.10	0.05	0.08	0.09	0.07	0.10
Temperature, °C	180	180	180	180	180—200	180—230	180—200	180—220
Pressure, atm.	3	2	2	2	about 2	1—1.8	about 2	2.2—2.8
Time, h	4	4	4	4	4.5	3.5	6.0	4.0
Analysis of the raw material								
IV	55	58	58	58	59	58	60	57
Analysis of product								
IV	0.7	2.6	0.5	4.1	7.5	3.5	7.5	1.4

fresh catalyst was distributed in the lard taken from the autoclave after 1 hour of the "preliminary hardening", whereas in the industrial scale process lard filtered out of the first filter-press was fed to the agitator of the fresh catalyst.

A comparison of the large-laboratory scale and industrial scale process shows that the scaling-up gives products with a slightly higher iodine values. These values however, are still within the limits up to 10 units. The similar situation takes place for the specified amount of the catalyst and for the time of the process. Both values are higher for the industrial process, but they are still within the specified limits. Hydrogen pressure is slightly lower than that in the laboratory process.

Table 2

Characteristics of the hardened technical grade lard

Iodine value (determined by the method of Hanus)	maximum 10
Titre, °C	minimum 55
Colour, mg I/100 ml	maximum 16

Characteristics of the hardened technical grade lard is given in Table 2.

Product of such characteristics can be used as a very good addition to the soap feed-stock.

Some examples of the hardening of the distilled mixed soap-stock fatty acids are given in Table 3.

These low grade acids characterized by the high ester value and also by the high unsaponifiable matter, ash and sulphur content contain erucic acid remaining from the processing of the rapeseed oil. High active catalyst must be used for the hardening of these acids. Amounts of the fresh catalyst necessary to obtain product with iodine value below 10 vary from 0.16 to 0.6% of Ni. Results obtained with the method presented hereby are shown in Table 4 in comparison with the typical method of the hardening of fatty acids, carried out solely in the presence of the fresh catalyst odded at 130°C to the fatty acids by which hydrogen has been already passed.

A comparison of both processes, carried out under the standard conditions, shows that the application of the preliminary hardening makes possible to reduce the consumption of the fresh catalyst. Futhermore recycling of the catalyst does not affect the possibility of its regeneration carried out to recover the nickel. Thus the reusage of the catalyst does not diminishof its usefulness for the regeneration process.

Table 3

Characteristics of the mixed vegetable soap-stock fatty acids distilled under the industrial scale conditions

Ester value		3
Iodine value		61
Colour, mg l/100 ml		35
Unsaponifiable matter, %		1.3
Water, %		0.2
Sulphur, mg/kg		10
Composition of the fatty acids, % by weight		
below	C_8	—
	$C_{8:0}$	—
	C_x	—
	$C_{10:0}$	—
	$C_{12:0}$	3.0
	C_x	—
	$C_{14:0}$	2.5
	$C_{14:1}$	—
	$C_{14:2}$	—
	C_{15}	—
	$C_{16:0}$	17.0
	$C_{16:1}$	1.7
	$C_{16:2}$	0.5
	C_{17}	0.5
	$C_{18:0}$	10.9
	$C_{18:1}$	31.2
	$C_{18:2}$	11.8
	$C_{18:3}$	3.3
	$C_{20:0}$	
	$C_{20:1}$	3.9
	$C_{22:0}$	—
	$C_{22:1}$	13.7

Table 4

Hardening of the distilled mixed soap-stock fatty acids carried out under the large-laboratory scale conditions

	I	II
Parameters of the process		
1st stage: Ni, %	—	0.07
Temperature, °C	—	175
Pressure, atm.	—	25
Time, h	—	1
2nd stage: Ni, %	0.12	0.08
Temperature, °C	175	175
Pressure, atm.	25	25
Time, h	4	4
Results of the analysis		
IV of raw material	77.3	72.5
IV of product	7.4	1.5

CONCLUSIONS

Two-stage process of the hardening of the technical grade fats and fatty acids improves the effectiveness of the batch process. By the application of the first stage of hardening of technical grade fats, carried out for 0.5-1.5 hour, at 150-170°C under the pressure of 2-5 atmposphere in the presence of the catalyst which has been already used in the similar process, and by the similar process, and by the distribution of the fresh catalyst used in the second stage in fat free of catalyst poisons, the 50% reduction of the catalyst consumption has been achieved.

In case of the hardening of fatty acids the 20% reduction of the catalyst consumption has been achieved. Process conditions for the first stage are similar as in the hardening of the technical grade fats. The only difference is the pressure, which is kept on the level of 20 atmospheres.

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SPOSÓB DODAWANIA SPROSZKOWANEGO KATALIZATORA W PROCESACH UTWARDZANIA KWASÓW TŁUSZCZOWYCH I TŁUSZCZÓW TECHNICZNYCH

Streszczenie

W procesie periodycznego utwardzania tłuszczów technicznych i kwasów tłuszczowych stosowane są wysokoaktywnekatalizatory proszkowe, które łatwo ulegają dezaktywacji już w okresie pierwszego zetknięcia z surowcem.

Wprowadzona modernizacja technologii polega na tym, że w I etapie do ogrzanego surowca dodaje się zużyty w analogicznym procesie katalizator i prowadzi się proces w czasie minimum 30 minut w odpowiednich warunkach. Następnie, w II

etapie, dodaje się świeży katalizator rozprowadzony w produkcie po pierwszej filtracji lub niefiltrowany.

Zachowanie wysokiej aktywności dodanego katalizatora pozwala na zmniejszenine jego zużycia oraz na uzyskanie niższych liczb jodowych w trudno utwardzających się surowcach.

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СПОСОБ ПРИБАВЛЕНИЯ ПОРОШКООБРАЗНОГО КАТАЛИЗАТОРА В ПРОЦЕССАХ ОТВЕРЖДЕНИЯ ЖИРНЫХ КИСЛОТ И ТЕХНИЧЕСКИХ ЖИРОВ

Резюме

В процессе периодического отверждения технических жиров и жирных кислот используются высокоактивные порошкообразные катализаторы, которые легко дезактивируются уже в период первого контакта с сырьем.

Введение модернизации технологии заключается в том, что в первом этапе к огретомусырью добавляют использованный в аналогичном процессе катализатор и проводят процесс на протяжении по крайней мере 30 минут в соответствующих условиях. Затем, во втором этапе прибавляют свежий катализатор распределенный в продукте после первой фильтрации или нефильтрованном.

Удержание высокой активности добавленного катализатора позволяет сократить его потребление и получать более низкие иодные числа в трудно отверждающемся сырье.