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STUDY OF WAYS TO REDUCE THE VISCOSITY OF SUNFLOWER LECITHIN

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Abstract. Today, lecithin is the most important by-product of oilseed processing due to its numerous technological properties. Food industry technologists use lecithin as an emulsifier, wetting agent, stabilizer, viscosity regulator, crystal formation, etc. One of the main disadvantages of sunflower lecithin is the tendency to increase viscosity (plasticization) during storage. Such lecithin is difficult to use. Therefore, obtaining liquid lecithin with a viscosity of less than 12 Pa s (at 25°C), which does not increase during its storage, is one of the urgent tasks of enterprises for the extraction and processing of vegetable fats. The article compares diluents of different types. Diluents of fatty nature - sunflower oil, oleic acid and its ethyl ester are characterized by a significantly lower viscosity compared to lecithin, therefore they can act as diluents that do not affect the quality of lecithin. However, their amounts are too large to achieve the goal of obtaining liquid lecithin. These amounts significantly reduce the "acetone insoluble" lecithin index. That is, oils, their acids and esters cannot be recommended for reducing the viscosity of lecithins in industry. It has been shown that the efficiency of divalent calcium salts as diluents is much greater, even at low concentrations of these substances. It has also been proven that the introduction of diluents must be carried out before the stage of drying the wet gum. To obtain liquid lecithin from plasticized lecithin, approximately twice as high concentrations of diluents are required compared to the preventive approach – the introduction of diluents into a wet gum. To obtain liquid sunflower lecithin from a wet gum to which diluents were added, the effective concentrations of diluents are as follows, %: sunflower oil – 13, oleic acid – 5, ethyl ester of oleic acid – 4, calcium chloride – 0.35, calcium acetate – 0.4, calcium orthophosphate – 0.4, concentrated phosphoric acid – 2. It is most expedient to use calcium salts in order to obtain high quality liquid lecithin.

Keywords: plastic lecithin, liquid (fluidize) lecithin, diluents, sunflower lecithin, wet gum.

Introduction. Formulation of the problem

The field of production and use of lecithins today is formed, but several factors affect its changes. Soybeans are the world's leading source of lecithin, but there is a growing demand for lecithin from other non-GMO sources, most notably sunflower and rapeseed. Although soy lecithin from GMO soy has been shown to be equivalent to GMO-free lines, the European market prefers non-GMO lecithin [1]. The discovery of liposomes has provided a new and more efficient way

to deliver drugs based on the use of lecithin. One of the components of lecithin – choline is one of the physiologically necessary food ingredients (vitamin B₄), so the increased demand for lecithin as a dietary supplement is expected [2]. All this expands the scope of sunflower lecithin, indicates an increase in demand and, probably, prices for it.

Analysis of recent research and publications

Most commercial lecithins are obtained by hydration of soybean extraction oil (due to the high

content of phospholipids in this raw material – about 1.8%). At the same time use 1–2% of water or steam at a temperature of 40–70°C [3]. The resulting wet gum (water content 25–50%) is separated by separators (separation by settling and decantation, of course, much less expensive process due to the high cost of separators, but takes more time and does not lead to clear phase separation). Bleaching agents and / or diluents are added to the wet gum before drying. The product obtained after drying (water content less than 1%) is called "unrefined" or "natural" lecithin (or phosphatide concentrate), with a content of phospholipids 60–70% and oil 30–35% [4].

On the basis of natural lecithin get a number of products (Fig. 1), which are characterized by slightly different technological properties and, therefore, field of application.

Lecithin performs the following main functions in foods [2,5-7]:

- emulsification – allows mixing and maintains the homogeneity of lipophilic and lipophobic components of food systems (margarine, mayonnaise and other sauces, chocolate, etc.). This is the most common use;
- solubilization – allows you to dissolve lipophilic substances in water (such as essential oils and fat-soluble dyes);
- dispersant – for example, retains pigments dispersed in paints, preventing their agglomeration;
- wetting agent – helps substances to dissolve quickly in water (instant soups, instant drinks, etc.);
- non-stick lubricant – thin films of lecithin are applied to the mold; lecithin promotes the release of food or other materials from this surface;
- control of crystallization – for example, sugar in fatty systems, in particular in chocolate;
- complexation – slows down the crystallization of starch, which is the main cause of hardening of bread,

which has a positive effect on the shelf life of bread, bakery products, etc.;

- anti-splatter (margarines, confectionery fats);
- viscosity change – widely used in chocolate production;
- antioxidant properties – lecithin inhibits the rate of oxidation of fats; it is a chelator of metals;
- therapeutic effect - indispensable for the growth, development and functioning of all somatic cells of the body, are antioxidants, participate in the nervous system, perform many other functions [8,9].

Thus, lecithin is a multifunctional food ingredient with a wide range of applications.

Different countries use different methods for studying the quality of lecithins, therefore, the requirements for them are different. Comparative analysis of the requirements for lecithins is summarized in table 1.

A significant difference is observed in the definition of such indicators as the mass fraction of mechanical impurities and color.

The two main problems of sunflower lecithins produced in Ukraine, which often do not allow to call them the international term "lecithin" – is, first, the possible high content of mechanical impurities (insoluble in hexane or insoluble in toluene, or insoluble in ethyl ether according to various standards countries); second, high viscosity. The problem of high content of mechanical impurities is solved by effective filtration of oil before refining (separators are used for this purpose). Therefore, there is no need to solve the first problem in this study. The decrease in viscosity is possible during the technological exposure to the lecithin, preferably during the drying stage of the lecithin. Even more appropriate is the introduction of phospholipid diluents at the stage of degumming of the oil.

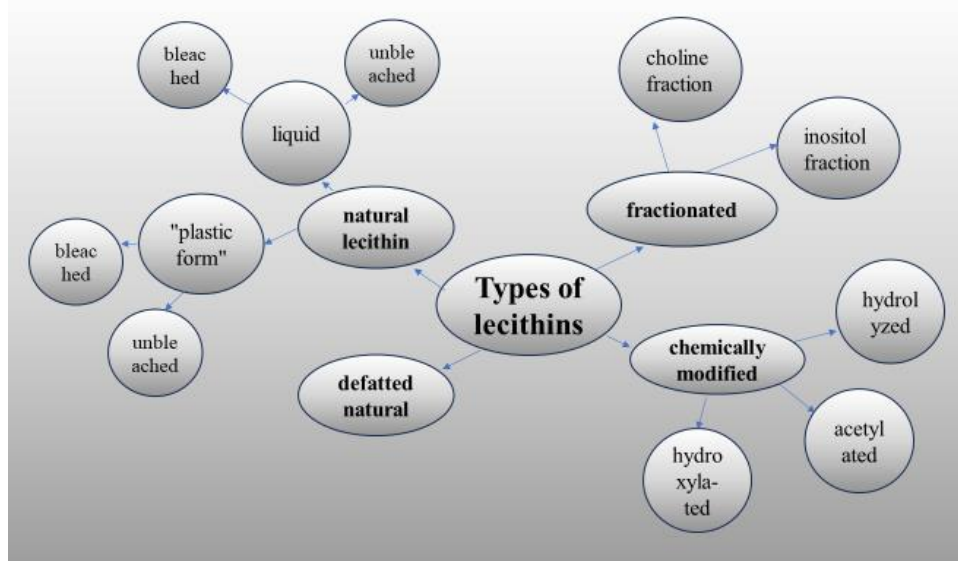


Fig. 1 Types of commercial lecithins

Table 1 – Requirements for lecithins

Indicator	SOU -15.4-37-212:2004 "Phosphatide concentrates. Specifications "	EU № 231/2012 (Lecithins 322)
Acetone-insoluble matter, %	60	60
Moisture and volatile matter content, %	1.0	Loss on drying. Not more than 2% (105°C, 1h)
Mass fraction of mechanical impurities	Ethyl ether insolubles. Not more than 1.5%	Toluene-insolubles. Not more than 0.3% (some firms use the indicator Hexane Insolubles $\leq 0.5\%$)
Acid value, mg KOH/g, not more than	Acid value of oil isolated from phosphatide concentrate, mg KOH/g, 10	not more than 36 mg of potassium hydroxide per gram
Peroxide value, mmol $\frac{1}{2}$ O/kg, not more than	Peroxide value oil isolated from phosphatide concentrate, not more than 10 mmol $\frac{1}{2}$ O/ kg	10
Viscosity at 25 °C, Pa·s, not more than	12	12
Color	Color, mg of iodine, not more than – 8 (1% solution in hexane)	Unit of Gardner scale – 17

The viscosity of sunflower lecithin should not exceed 12 Pa·s (at 25°C). Most often, enterprises produce soy lecithins of significantly higher viscosity – high-viscosity, often no longer fluid, the so-called «plastic forms». Such lecithins must be melted for transportation and use. It is inconvenient for technologists to work with them. In addition, more fluidize lecithins are more easily distributed in food and perform their functions better. Therefore, most consumers of lecithins want to get a product with a viscosity of 8–12 Pa·s.

Fluidize lecithins usually correspond to Newtonian characteristics. The value of viscosity is influenced by the content of phospholipids, moisture content, metal content. Increasing the content of phospholipids and moisture increases the viscosity. The presence of metals reduces the viscosity of lecithins [10].

Soy lecithin has been found to have the lowest viscosity compared to rapeseed and sunflower lecithin. The high viscosity of sunflower lecithin is due to the presence of long-chain waxes [2].

The wet gum in the drying process changes from a Bingham-type liquid (continuous water) to a

pseudoplastic liquid (continuous phase). The maximum viscosity occurs in the range of 6.9–19.3% moisture content, which complicates the process of drying lecithin in a thin film [11]. The unique rheology of the lecithin-water system has a major impact on the difficulties in drying lecithin. Dried lecithin, even at low moisture content, exhibits significant viscoelastic behavior, to an extent not usually associated with low molecular weight polymer chains. This is due to the formation of an electrostatic gel, which occurs due to the electrostatic nature of the polar part of the lecithin molecule, the charged nature of the micelles and the significant dipole moment of phospholipid molecules. Wet gum consists of approximately 35% phospholipids, 50% water and 15% oil. Phospholipids are located between the polar phase (water) and non-polar (oil) in the form of a two-layer micellar structure [11].

To reduce the viscosity of phosphatide concentrates, the simplest option is to use a solution of sodium chloride, which is introduced at the stage of drying the phospholipid emulsion. Sodium chloride not only reduces the viscosity of lecithin, but also allows less traumatic for lecithin to carry out the drying process (lighter color, less soot, etc.) [12]. However, the presence of salt in lecithin can affect the content of mechanical impurities (salt is insoluble in ethyl ether or hexane), so it is advisable to use other diluents.

An alternative solution to the problem of high viscosity of phosphatide concentrates is to introduce other lipids with low viscosity. Mono-, diglycerols of vegetable oils or special oils with fatty acids with a chain length of C12-C16 are added to the phosphatide concentrate. Another way is the introduction of methyl or ethyl esters of fatty acids [13].

In our opinion, it is optimal to use substances that, when administered in small quantities, can significantly reduce the viscosity of lecithin – for example, salts of divalent metals [13]. Trivalent metal ions, such as aluminum, also have a pseudo-liquefying effect; however, they tend to increase the acid number, ie it is necessary to neutralize lecithin, after the addition of alkali plasticization of lecithin occurs again.

The purpose was to compare the effectiveness of substances that can reduce the viscosity of lecithins to values of ≤ 12 Pa·s (at 25°C).

Objectives of the research:

1. Improving the quality of sunflower lecithins by reducing their viscosity when using safe fatty substances with low viscosity - sunflower oil, oleic acid or its ester.

2. Study of divalent metal salts as lecithin diluents.

3. Study of the influence of optimal diluents on other indicators of the quality of lecithin. Comparison of the effectiveness of diluents when they are introduced into a wet gum or into already plasticized lecithin.

Research materials and methods

Sunflower lecithin with a phospholipid content of 65.5% was provided by a Ukrainian manufacturer, as was a sunflower wet gum of the following composition: water – 65.5%, phospholipids – 19.7%, neutral lipids – 12.1%.

Sunflower lecithin was heated to a temperature above its melting point – 60°C, added certain amounts of sunflower oil or oleic acid or ethyl ester of oleic acid. At a temperature of 60°C (water bath) was carried out homogenization using a propeller stirrer (120 rpm) for 10 minutes. The resulting mixture was cooled to room temperature and stored for 14 days. Viscosity was then determined.

To the sunflower wet gum were added certain amounts of diluents, at a temperature of 60°C was stirred for 10 minutes and dried the wet gum at 80°C in a rotary film evaporator under reduced pressure to a moisture content of <1%. The obtained lecithin was cooled to room temperature and stored for 14 days at low temperatures – 5–10°C (for the fastest plasticization). Viscosity was then determined.

In the studies used 30% solutions of calcium chloride, calcium acetate, calcium orthophosphate.

Viscosity was determined by Brookfield Viscometer LVT (USA) with eight speeds: 0.3, 0.6, 1.5, 3, 6, 12, 30 and 60 r.p.m. The viscosity of lecithin was determined at 25°C by immersion in a thermostatic bath (TC-1/80 CИY). Viscosity is measured by converting the torque required to rotate the instrument's spindle at a constant speed when it is immersed in the test medium.

Qualitative indicators of lecithins were studied according to SOU 15.4-37-212: 2004 "Phosphatide concentrates. Specifications".

All experimental data were obtained twice. The article gives average values. The mean deviation did not exceed 0.1 for a confidence interval of 0.95.

Results of the research and their discussion

Most lecithins are fluidize immediately upon receipt, but are prone to plasticization as a result of storage. Initially, it was decided to reduce the viscosity of sunflower lecithin containing phospholipids (62.8%). Diluents were added to the wet gum and dried, trying to approach the moisture level of 0.7% – with this amount of water, lecithin is most likely to plasticize [13]). The results of studies on the reduction in the viscosity of sunflower lecithin (after 14 days of storage) when sunflower oil or fatty acids or ethyl esters are added to it are shown in fig. 2. Thus obtained sunflower lecithin without the addition of diluents acquired a plastic consistency.

At the same time, all the above diluents were added in the same amounts to sunflower lecithin obtained from the manufacturer and already plasticized. It was possible to achieve a reduction in viscosity to a liquid state of lecithin only by adding either 25% oil, or 11% oleic acid, or 10% ethyl esters. There is a significant difference between the efficiency of adding diluents before drying lecithin (into a phospholipid emulsion, data in Fig. 2) and in already plasticized lecithin. It is necessary to draw a conclusion about the advisability of adding diluents before the stage of drying the phospholipid emulsion. In further studies, only phospholipid emulsion was used.

The use of oil as a substance that reduces the viscosity of lecithin can not be recommended for use in industry – the effect of reducing viscosity to the required 12 Pa·s occurs at $\approx 13\%$ of added oil, ie at 44% of total neutral fats in lecithin. The amount of acetone-insoluble substances (actually phospholipids), thus less than 60%, which is unacceptable in the production of competitive natural lecithin.

Fatty acids have a significantly lower viscosity than lecithin and oil. The use of oleic acid in the amount of 5% corresponds to a decrease in viscosity to the requirements of regulatory documents 12 Pa·s. However, this is accompanied by a significant increase in the acid number and a decrease in the content of acetone-insoluble substances in phosphatide concentrate – from 62.8 to 60.4%, ie almost the limit value according to regulatory requirements. Thus, the use of oleic acid is acceptable, but obviously not the best option.

The addition of ethyl esters leads to a decrease in the viscosity of the content of the latter indicator. Achieving 12 Pa·s for the studied lecithin corresponds to the introduction of 4% ethyl ester of oleic acid. Ethyl esters have proven to be the most effective in reducing the viscosity of lecithin, but they are quite expensive substances, 4% of the introduced ether again underestimates the content of phospholipids. Therefore, it is necessary to investigate the effect of other diluents, which to achieve the goal is sufficient to enter in significantly smaller quantities. These include divalent metal ions [13]. It was decided to study only calcium derivatives as an element that is vital for the human body [14]. The research results are shown in Fig. 3.

Calcium chloride (E 509) is the most common of the lecithin diluents on the world market. Recognized as highly effective and safe [15]. Its presence in the amount of approximately 0.35% is sufficient to reduce the viscosity of sunflower lecithin (plasticized without the addition of diluents) to a value of 12 Pa·s.

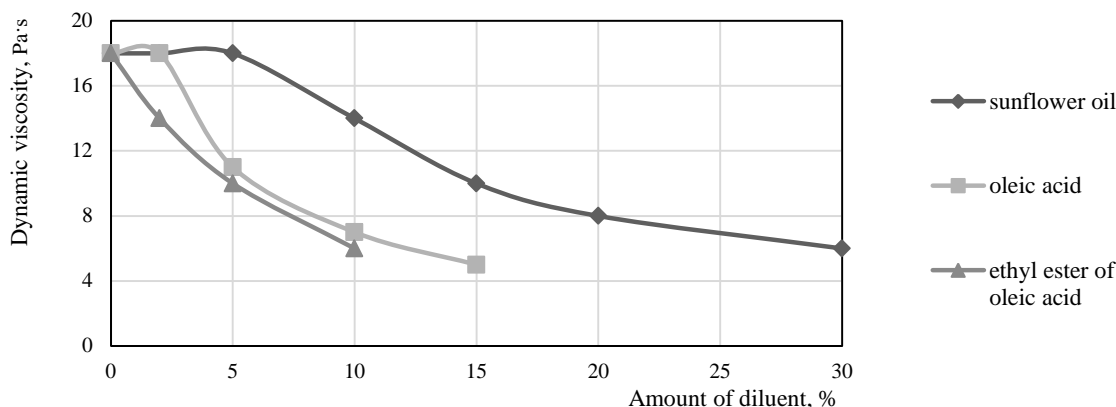


Fig. 2 Effect on the dynamic viscosity of sunflower lecithin sunflower oil, oleic acid, ethyl ester of oleic acid (values obtained at 25°C)

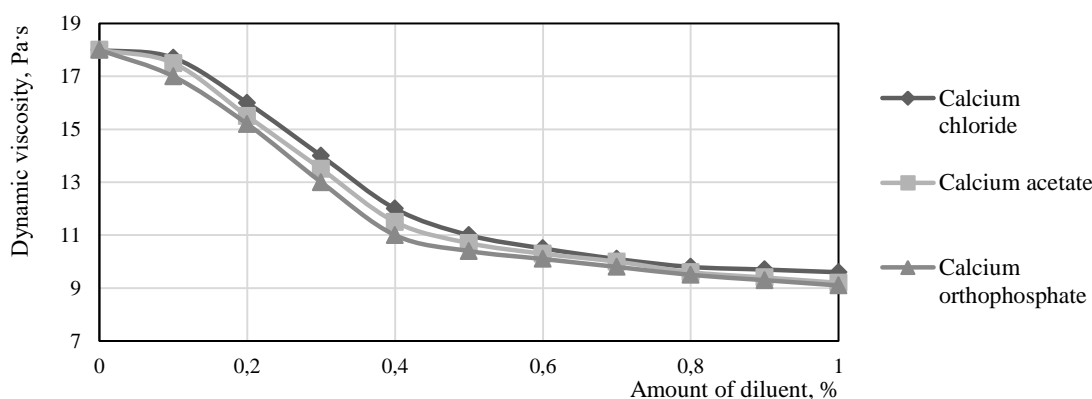


Fig. 3 The effect of divalent metal salts on reducing the viscosity of sunflower lecithin

Calcium acetate or calcium acetate (general formula $\text{Ca}(\text{CH}_3\text{COO})_2$) is registered as a food additive E 263 (preservative, acidity regulator). Permissible daily consumption is not limited, which indicates the safety of this substance. Acetates are normal components of the human and animal diet, produced in the gastrointestinal tract. They are completely metabolized in the human body [16]. Calcium acetate is used in pharmaceuticals to prevent bone loss and improve bone microarchitecture [14]. At an amount of $\leq 0.4\%$ of calcium acetate, fluidize sunflower lecithin is obtained (dynamic viscosity is 12 Pa·s).

The use of calcium atrophosphate ($\text{Ca}_3(\text{PO}_4)_2$) is appropriate for the dilution of lecithin. It contains about 40% of elemental calcium (which actually reduces the viscosity of lecithins). The other is the PO_4^- group, which is found in all phospholipids without exception. That is, when determining the content of phospholipids in lecithin by the most common method, which determines the content of phosphorus proper (AOCS Ca12-15), the presence of calcium phosphate will be almost invisible in terms of quantitative composition of lecithin. Calcium orthophosphate is used as a food additive E341 (stabilizer, acidity regulator, color fixative, baking powder). In the human body, calcium exists mainly in the form of calcium phosphates. 70% of human bones are composed of calcium phosphates [17]. Combined

with the low price, these useful characteristics of calcium orthophosphate make it a promising diluent, effective in the amount of $\leq 0.4\%$.

It was further decided to reduce the viscosity of sunflower lecithin with a high content of phospholipids (65.5%), which has been stored for some time and acquired a plastic consistency. The results of studies to reduce the viscosity of soy lecithin (after 14 days of storage from the date of addition of diluents) are given in table 2. Namely, the results of studies with established rational amounts of diluents, which lead to a decrease in the viscosity of soy lecithin to a value of 12 Pa·s (at 25°C).

Table 2 – The amount of diluents that reduce the viscosity of sunflower lecithin to 12 Pa·s (at 25°C)

Diluent	Quantity, % in relation to soy lecithin
Soybean oil	25 ± 0.6
Oleic acid	11 ± 0.5
Ethyl ester of oleic acid	10 ± 0.5
Calcium chloride	0.7 ± 0.06
Calcium acetate	0.8 ± 0.08
Calcium orthophosphate	0.8 ± 0.06
Concentrated phosphoric acid	2 ± 0.2

As can be seen from the data in the table. 1 the amount of diluents required to return the already plasticized lecithin to a liquid state is approximately twice the amount of appropriate diluents, which is sufficient to obtain liquid lecithin from the wet gum to which these diluents were added. Also, in our second study, it was shown that some of the diluents are effective degumming agents, that is, they increase the yield of phospholipids from vegetable oil during the degumming process [18]. That is, calcium salts should be used already at the stage of formation of a wet gum, while the quality of future lecithin and oil improves.

Concentrated phosphoric acid in the amount of 2% is an effective diluent. However, under its influence there is a darkening of lecithin (to a value of about 14 mg of iodine) and a sharp increase in the acid number (from 9.5 mg KOH to 20.3 mg KOH). Therefore, in our opinion, the use of concentrated acids as diluents should be abandoned.

The effect of reducing the viscosity to the required 12 Pa·s occurs at 25% of added soybean oil, ie at 50% of the total content of neutral fats in lecithin. The amount of acetone-insoluble substances (actually phospholipids), thus less than 60%, which is unacceptable.

Approximately 11 and 10% oleic acid or its ester is sufficient to obtain fluidize lecithin soy lecithin. This is accompanied by the above shortcomings.

Calcium ions have proven to be the most effective diluents. Calcium chloride, acetate, orthophosphate in amounts $\leq 0.8\%$ make it possible to obtain fluidize soy lecithin with a viscosity of ≤ 12 Pa·s.

In order to establish the influence of the most effective diluents on the quality of sunflower lecithin, the latter's compliance with the requirements of the current SOU 15.4-37-212: 2004 "Phosphatide concentrates. Specifications" was determined (Table 2). Sunflower lecithin was obtained by drying a wet gum to which calcium orthophosphate was added in an amount of 0.4%.

Sunflower lecithin obtained by drying a wet gum containing 0.4% calcium orthophosphate meets all the requirements of the current standard. The presence of calcium orthophosphate in lecithin did not negatively affect such important indicators of lecithin as peroxide number, acid number and color. It is especially important that the presence of this salt did not have a negative effect on the indicator "mass fraction of substances insoluble in ethyl ether" (equal to 0.6%, Table 3). This indicator is very sensitive to the introduction of salts and other substances, and its low value is probably related to the finely dispersed state of calcium orthophosphate and its partial dissociation into PO_4^{3-} and Ca^{2+} groups in lecithin.

Table 3 – Qualitative indicators of sunflower lecithin (amount of calcium orthophosphate 0.4%)

Indicator	The resulting sunflower lecithin	Requirements of SOU 15.4-37-212: 2004 "Phosphatide concentrates. Specifications"
Mass fraction of substances insoluble in acetone, %	62.8 ± 0.09	≥ 60
Mass fraction of moisture and volatile substances, %	0.73 ± 0.05	< 1.0
Acid value of oil isolated from phosphatide concentrate, mg KOH/g	5.6 ± 0.11	< 35
Peroxide value, mmol $\frac{1}{2}\text{O}_2/\text{kg}$	2.8 ± 0.18	≤ 10
Mass fraction of substances insoluble in ethyl ether, %	0.6 ± 0.10	≤ 1.5
Viscosity at 25°C, Pa·s	10 ± 0.20	≤ 12
Color, mg of J_2	6 ± 0.50	≤ 8

Conclusion

1. Nowadays, it is believed that sunflower lecithins are inferior to soy in terms of color, taste and texture. Reducing the viscosity of sunflower lecithins is one way to solve the problem of their full entry into the world market. The introduction of neutral oil, fatty acids, esters of fatty acids leads to the dilution of lecithin to values below 12 Pa·s (at 25 °C). However, in order to achieve a significant reduction in the viscosity of lecithins, these lipids must be administered in significant amounts, which reduces the value of the indicator "insoluble in acetone" to a level below the requirements of regulations. Also, when using these diluents, lecithin stratification often occurs later – the separation of some neutral lipids from polar. Under the conditions of their introduction to the wet gum, the following amounts are sufficient: sunflower oil – 13%, oleic acid – 5%, oleic acid ethyl ether – 4%. The use of concentrated (98%) phosphoric acid in the amount of 2% leads to a decrease in the viscosity of lecithin ≤ 12 Pa·s. However, this is accompanied by a significant increase in the acid number of lecithin (from 9.5 mg KOH to 20.3 mg KOH). Under the influence of phosphoric acid there is a darkening of lecithin (from 6 to 12 mg of iodine). That is, the quality of lecithin deteriorates markedly. Thus, the use of concentrated strong acids cannot be recommended as a method of diluting lecithin.

2. Calcium salts turned out to be the optimal diluents as a result of research. The effective amounts of their presence for the production of fluidize lecithin with a viscosity of ≤ 12 Pa·s were 0.35–0.5% relative to lecithin (in the conditions of introduction before drying the wet gum). Increasing the content of divalent metals above 0.5% practically does not lead to a further decrease in viscosity. Calcium salts allow to obtain high quality lecithins.

3. Sunflower lecithin obtained by drying a wet gum containing 0.4% calcium orthophosphate meets all the requirements of the current standard. The addition of diluents to lecithin should be carried out before the drying stage due to the positive effect of reducing the viscosity of the drying process. Also, the effective amounts of diluents needed to obtain liquid lecithin from already plasticized lecithin are approximately twice the amount of the corresponding diluents added at the stage of drying the wet gum.

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БІОХІМІЧНІ ОСОБЛИВОСТІ НАСІННЯ СОРТІВ ЛЬОНУ ОЛІЙНОГО СПЕЦІАЛЬНОГО ПРИЗНАЧЕННЯ

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Анотація. Лецитин сьогодні є найбільш важливим побічним продуктом перероблення олійних через його численні технологічні властивості. Технологи харчової промисловості використовують лецитин як емульгатор, змочувач, стабілізатор, регулятор в'язкості, кристалоутворення тощо. Одним з головних недоліків соняшникового лецитину є схильність до підвищення в'язкості (пластифікування) в процесі зберігання. Такий лецитин важко застосовувати. Тому одержання рідкого лецитину з в'язкістю менше 12 Па·с (при 25°C), яка не збільшується в процесі його зберігання – одна з актуальних задач підприємств з добування та перероблення рослинних жирів. В статті порівнюються розріджувачі різних типів. Розріджувачі жирової природи – соняшникова олія, олеїнова кислота та її етиловий ефір характеризуються суттєво нижчою за лецитин в'язкістю, тому можуть виступати розріджувачами, що не впливають на якість лецитину. Однак їхні кількості є занадто великими для досягнення мети – одержання стабільно рідкого лецитину. Ці кількості суттєво знижують показник лецитину «нерозчинні в ацетоні». Тобто олії, їх кислоти та ефіри не можна рекомендувати для зниження в'язкості лецитинів у промисловості. Показано, що ефективність двовалентних солей кальцію як розріджувачів є суттєво більшою, навіть при невеликих концентраціях цих речовин. Також доведено, що введення розріджувачів необхідно проводити перед стадією сушіння фосфоліпідної емульсії. Для одержання рідкого лецитину з вже пластифікованого необхідно приблизно вдвічі більші концентрації розріджувачів порівняно з превентивним підходом – введенням розріджувачів у фосфоліпідну емульсію. Для одержання рідкого соняшникового лецитину з фосфоліпідної емульсії, до якої були додані розріджувачі, ефективні концентрації розріджувачів є наступними, %: соняшникова олія – 13, олеїнова кислота – 5, етиловий ефір олеїнової кислоти – 4, хлорид кальцію – 0,35, ацетат кальцію – 0,4, ортофосфат кальцію – 0,4, концентрована фосфорна кислота – 2. Найбільш доцільно з метою одержання рідкого лецитину високої якості застосовувати солі кальцію.

Ключові слова: пластифікований лецитин, рідкий лецитин, розріджувачі, соняшковий лецитин, фосфоліпідна емульсія.