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Side streams from the vegetable oil production as feedstock for surfactants and their derivative technical systems

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This work conducts a technical analysis of the current production and consumption of vegetable oils and some of the oily byproducts to determine the potential feedstock for the synthesis of non-food-competitive surfactants and surfactant-based systems. It defines the concentrated phosphatides (phosphatidic sludge) as no-/low- value streams, appropriately suited for chemical valorisation. The study further creates biobased surfactants by amidation of phosphatidic sludge derived from refinery of sunflower and rapeseed oils with monoethanolamine, N-(2-hydroxyethyl)ethylenediamine, or N,N'-bis(2-hydroxyethyl)ethylenediamine, under the action of calcium hydroxide as catalyst in excellent yields (95–98 %). Besides waste remediation, the use of phosphatides enabled to create the mixed surfactant compositions, comprising fatty acid alkanolamides and calcium glycerolphosphatides with improved solubility in organic non-polar solvents. With new surfactants, there have been created reversed emulsion systems, which can be potentially applied to the development and exploitation of gas and oil deposits are recommended for drilling wells, the disclosure of productive strata; perforation of wells and development of productive layers; blockage of gas, gas condensate and oil wells; elimination of manifestations and flow of gas in wells; limitation and elimination of waterways; cleaning of the hollow zone of wells and intensification of the inflow of hydrocarbon raw materials, which have been tested in laboratory and experimental industrial conditions, and a significant part of them have been introduced or tested on gas condensate fields. Creation of coordinated, effective and economical actions that should be formed in the state energy policy of Ukraine would facilitate the development of oil and gas companies, namely: increase of own oil and gas production; maximizing the potential of energy saving; diversification of external sources of supply; approximation of the parameters of the oil and gas industry to the norms and standards of the European Union.

Keywords: : phosphatidic sludge, organic synthesis, surfactants, reversed emulsions.

Introduction

There have been manifold technical systems in the market, specifically designed to intensify extraction of crude oil and natural gas. They vary by origin (petrochemical- or plant-based), chemical composition, functional abilities, and market cost. To date, the prevailed quantities of technical products are made from petrochemical feedstock, of which toxicity and low biodegradability there is a dramatic environmental change. In view of the reduction of the environmental change, there is a want to develop technical systems, such as technical emulsions and suspensions, based on environmentally friendly, renewable feedstock. When it comes to the production of surfactants, and surface-active formulations, the major feedstock for biomanufactory remains vegetable oils; these products are less hazardous and are considered to be renewable [1,2]. The problem is that such surfactants are mostly produced from refined edible oils that compete with the food supply chain. In the present study, the focus is on the valorisation of non-food-competitive side streams from vegetable oil production and consumption that are compatible with ecological requirements to technical systems but which have only few commercialisable applications.

The works [1–5] demonstrated that the use of byproducts from vegetable oils manufacturing and secondary animal fat streams to produce technical surfactants could be alternative to the commonly applied refined substrates, such as rapeseed, sunflower, or corn oils. For example, in earlier works, there have been adopted varied vegetable oils to the synthesis of surfactants and invert dispersed systems (i.e. polar dispersed phase in non-polar bulk phase) thereof for their application in the exploitation of crude oil wells, as well as for the total well reconstruction [1]. Besides this and few other studies [1, 6, 7], to the best of our knowledge, there is limited or no information that relates to the synthesis and properties of commercially applied plant-based emulsifiers and their physicochemical properties from unrefined or waste lipid sources. These warrant an exploration of the synthesis methods and the availability of the feedstock to uncover potential ways to manufacture non-food-competitive plant-based surfactants. This work addresses this issue.

Which synthesis method is the best? Among the variety of synthetic approaches, the amidation of vegetable oils allows to produce a unique group of surfactants, namely fatty acid amides. These amides possess the high surface activity and create very dense

surface mono-, and sometimes poly-layers, of the strong N–H...O=C hydrogen bonding between amide groups [8, 9]. Such assembly between phase surfaces provides a variety of functions, for example, colloidal stability of emulsions [10]. The production of FAA from refined oils is not new and can be frequently met in literature. As a case in point, the amidation can be performed under acid- or base-catalysed conditions using alkanolamines as nucleophiles [11, 12]. With these versatile methods, there have been many surfactants synthesised from rapeseed oil [11–13], and herein, it is anticipated that similar techniques can be applied to convert non-food-competitive lipid substrates.

Which type of non-food biomass is the best?

While it is predictable that common synthesis practice, such as amidation, can be adopted in the production of surfactants from most lipid feedstocks, it remains unclear which substrates would be the most sustainable. Therefore, the total analysis of the harvesting of lipid crops, along with accompanying generation of the undervalued streams is required and is represented hereinafter with a focus on Ukraine.

In 2020, the gross production of oil crops in Ukraine decreased by 2,537 thousand tons (from 24.2 million tons in 2019 to 21.663 million tons in 2020) with the use of almost similar areas of arable lands (~8.885 million ha in 2019 and 8.809 in 2020). From the total harvest, rapeseed seeds accounted for 2.62 million tons, sunflower seeds for 16.272 million tons, and soybean seeds for 2.771 million tons, from which 2.42, 6.88, and 1.67 million tons oils, respectively, were exported to the countries of the European Union. For Ukrainian national consumption, 100 thousand tons of sunflower seeds were allocated for technical application and 50 thousand tons for food use. It is estimated that the 2020 harvest of sunflower simultaneously generated 440–580 thousand tons of low value streams, including residues after sedimentation and aqueous treatment, leading to the formation of phosphatidic sludge (PS, mixtures of phosphatides and vegetable oils) [14–18]. These streams can potentially be effectively employed in the

production of non-food-competitive technical surfactants, simultaneously strengthening the Ukrainian national economy. In this work, this potential is researched in the production of varied surface-active fatty acid alkanolamides (FAA). These new surfactants are further deployed in technical emulsions to test their operational abilities.

Materials and Methods

Materials. PS from the production of sunflower and rapeseed oils was supplied from Zaporizhian Oil and Fat Combinat, as per DSTU 4525. These materials comprise triacylglycerides with saturated (palmitic, stearic) and unsaturated (oleic, linolic, linoleic, gadoleic, erucic) fatty acid chains, along with phospholipids, such as phosphatidylcholine, phosphatidylethanolamine, phosphatidylserine, phosphatidylinositol, and phosphatidic acids. When received, these products were additionally dried under reduced pressure (5–10 mbar) to remove residual water and avoid any potential side reactions during storage. The dehydrated product appeared as viscous dark-brown liquids, named as PSs (derived from sunflower oil) and PSr (derived from rapeseed oil). Composition, including fatty acid composition, of PSr and PSs are represented in Table 1. All other materials, including monoethanolamine (MEA), *N*-(2-hydroxyethyl)ethylenediamine (HED), *N,N'*-bis(2-hydroxyethyl)ethylenediamine (BHED), calcium hydroxide, and diesel oil (for the emulsion preparation), were used as received from commercial sources.

Analytical methods. IR spectra were recorded on the IR-spectrometer Shimadzu IRAffinity-1Sn with ATR-console Speacac GS 10801-B. Differential thermal analysis (DTA), differential thermogravimetry (DTG), and thermal gravimetry (TG) were performed employing Derivatograph Q-1500D in a temperature range 20–500°C and heating rate 10 °C·min⁻¹. Operating characteristics of reversed emulsions were established according to standard procedures given in ПД 39-2-645-81. All other technical properties were measured using standard laboratory methods.

Table 1. Composition of PS from sunflower (PSs) and rapeseed (PSr) oils

Content	PSs	PSr
Phospholipids, %	51.7	50.5
Oil, %	46.8	48.2
Components insoluble in diethylether, %	1.2	1.1
Water, %	0.3	0.2
Fatty acid composition (acyl residue), %		
C _{16:0} (palmitic)	3.7	1.9
C _{18:0} (stearic)	4.7	1.8
C _{18:1} (oleic)	2.3	15.3
C _{18:2} (linoleic)	40.9	21.5
C _{18:3} (linolenic)	25.9	7.0
C _{20:1} (gadoleic)	2.2	2.1
C _{22:1} (erucic)	0.3	50.4

General methods. The synthesis of FAA was conducted employing base-catalysed amidation of PSs and PSr by MEA, HED, or BHED. Briefly, the syntheses were conducted by reactions of known amounts of PS (0.1 mol) and alkanolamines (0.3 mol) under the action of calcium hydroxide catalyst (40 mol%, based on PS). The reaction systems were introduced to the round-bottom flask equipped with reflux condenser and mechanical stirrer and were heated under constant agitation at 80–100 °C for 0.5 h and at 110–125 °C for 2.5 h. Reaction progress was monitored by potentiometric titration of amines, and by IR-analyses of the probes of the reaction media during the course of the processes. After syntheses, the unreacted amines were removed at elevated temperature (60–80 °C) under reduced pressure (5–10 mbar) in the nitrogen flow. As described earlier [19,20], calcium hydroxide forms complexes with phosphatides; therefore, any recovery of the catalyst or further purification was considered. FAA were named according to the source of PS and amine used for their production (PS-amine).

Preparation of reversed emulsions. To research emulsifying and stabilising properties of synthesised products, they have been used in the preparation of reversed emulsion systems. These systems can potentially be applied as technical fluids for oil well exploitation [1]. All emulsions were prepared by mixing known amounts of surfactants (0.1–2.5 wt%, based on the emulsion), mineralised water (a mixture of deionised water and calcium chloride; 40 and 7 wt%, based on the emulsion, respectively) and diesel oil (50.5–52.9 wt%, based on the emulsion) for 2 minutes at agitation rate 5,000 rpm.

Results and Discussion

Syntheses results are represented in Table 2, from which it is certain that reactions under selected conditions almost reached their completion, providing high yields of targeted FAA (96–98 %). There was noted that the use of BHED provided somewhat lower yields, likely associated with lower reaction rates of secondary amines. However, this should not be considered a disadvantage as the

targeted products and unreacted PS, apparently in the form of mono- or diacylglycerides, similarly possess high surface activity, which can be beneficial for varied application needs of mixed surfactants. This is one of the reasons why the proposed synthesis method avoids any refining of the product but the distillation of unreacted amines.

In earlier works [19, 20], there was a notion that alkaline metal hydroxides led to the formation of insoluble in organic solvents fractions of glycerolphosphatides. Follow-up solubility tests presented that all products are soluble in aliphatic (pentane, hexane, octane, petroleum ether), aromatic (toluene, xylene), and chlororganic (dichloromethane, chloroform) solvents without leaving any insoluble portions. These results suggest the interaction of calcium hydroxide, the catalyst, and residual glycerolphosphatides, which complexes are known to be oil-soluble materials [19, 20]. However, the exact nature of calcium-phosphatides complexes remains to be established.

The IR analysis identified the formation of FAA (Figure 1). In IR spectra of the products (Figure 1), new characteristic bands at 3300 cm^{-1} (N–H stretching vibrations), 1640 cm^{-1} (C=O amide stretching vibrations), and at 1560 cm^{-1} (N–H amide bending vibrations) appeared after reactions of PS and alkanolamines. At the same time, the characteristic vibrations of ester bonds at 1740 cm^{-1} (C=O ester stretching vibrations) disappeared after syntheses, additionally providing evidence in favour of the generation of FAA [19–21].

There have been also noted changes at 3600–3000 cm^{-1} (O–H stretching vibrations) and at 1050 cm^{-1} (P–O stretching vibrations of POOH) [21], likely associated with the interaction of phosphatides and calcium hydroxide. Considering the combined data, it is proposed the reaction course, as pictorially represented in Scheme 1.

Table 2. Synthesis of FAA from PS and their technical properties

Product	Yield, wt%	Consistence	Acid value, mg KOH/g	Fluidity temp, °C
PSs-MEA	98	<i>Solid</i>	0.90	59
PSs-HED	98	<i>Solid</i>	0.09	55
PSs-BHED	96	<i>Solid</i>	0.09	53
PSr-MEA	98	<i>Solid</i>	0.81	55
PSr-HED	97	<i>Solid</i>	0.06	53
PSr-BHED	95	<i>Solid</i>	0.06	51

PSs and PSr = phosphatidic sludge derived from sunflower and rapeseed oils, respectively. MEA = monoethanolamine, HED = *N*-(2-hydroxyethyl)ethylenediamine. BHED = *N,N'*-bis(2-hydroxyethyl)ethylenediamine. Reaction conditions: PS (0.1 mol), alkanolamines (0.3 mol), calcium hydroxide (40 mol %, based on PS), 80–100 °C, 0.5, and then 110–125 °C, 2.5 h

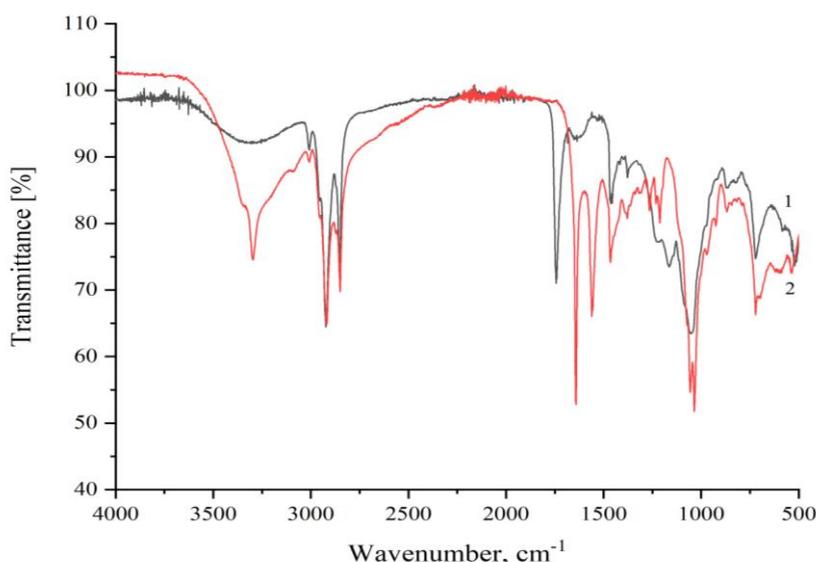
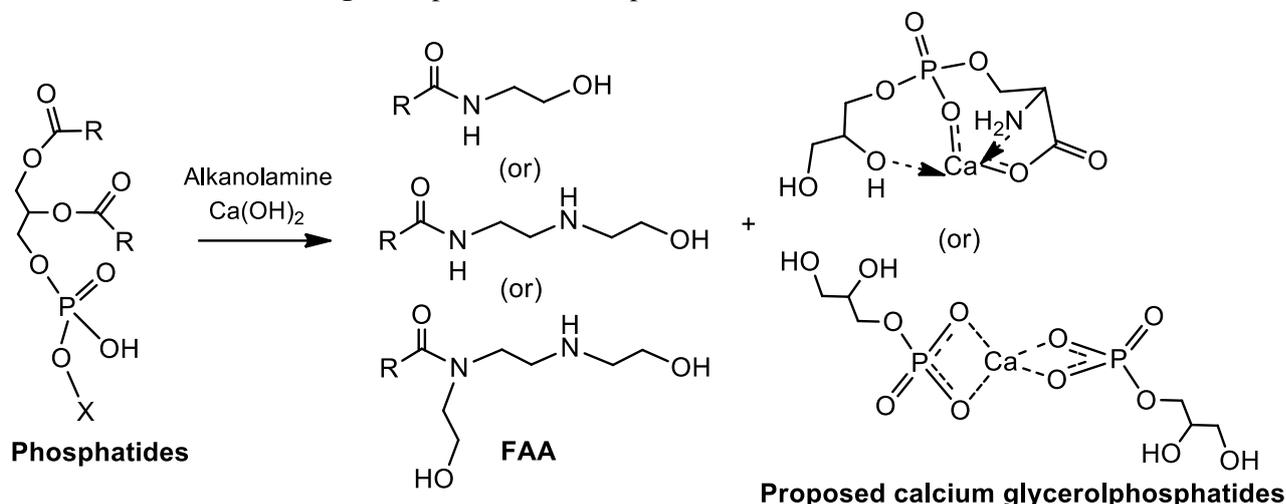


Fig. 1. Representative IR spectra of PS (1) and FAA (2).



Scheme 1. Proposed conversion of phospholipids into FAA. R = alkyl or alkenyl fatty acid residues. X = H or serine residue. Alkanolamine = monoethanolamine, *N*-(2-hydroxyethyl)ethylenediamine, or *N,N'*-bis(2-hydroxyethyl)ethylenediamine

DTA and DTG analyses revealed the extend of the thermal stability of FAA obtained from PS (Figure 2). DTA curve fixed three exothermic transitions at 40, 150 and 214 °C (Figure 2), which are accompanied by the loss of weight of FAA (Figure 2). Specifically, the weight loss started at 91 °C that can be considered a dehydration process, for example, through the removal of water from the surface-active amide groups [21]. The dehydration is accounted for 5% of the total weight losses. Further mass losses occurred at 181, and 336 °C (95% of total weight losses) and are likely attributed to the thermal decomposition of the product in several steps, which mechanisms are not yet clear. Such observations were noted for all produced FAA. From these results, it is deemed that synthesised amides can be exploited in technical processes at temperatures up to 180 °C; this high is a reasonable borderline in the surfactant application chemistry [1,2, 22-23].

Finally, all synthesised products were deployed in

the production of reversed (water-in-oil) emulsions. In an attempt to research a minimum required concentration of newly made surfactants to stabilize emulsions, the starting concentration of FAA was 0.1–0.25 wt%, based on emulsion; this is close to the critical micellar concentration for this class of surfactants.

However, such amounts were low for adequate stabilisation of the water/diesel oil emulsions and led to the speedy phase separation. Pleasingly, a follow-up increase of the surfactant concentration to 1.5–2.5 wt% improved the stability of emulsions, leading to the formation of highly stable dispersed systems (>90 days, Table 3). Noteworthy, the emulsions with new FAA remain stable even after extended storage at elevated temperature (80 °C) and in the conditions of the electrical stability tests (Table 3). These results shine the promise on the potential application of PS-derived FAA in varied drilling fluids during exploitation of crude oil and gas wells under forcing operation parameters.

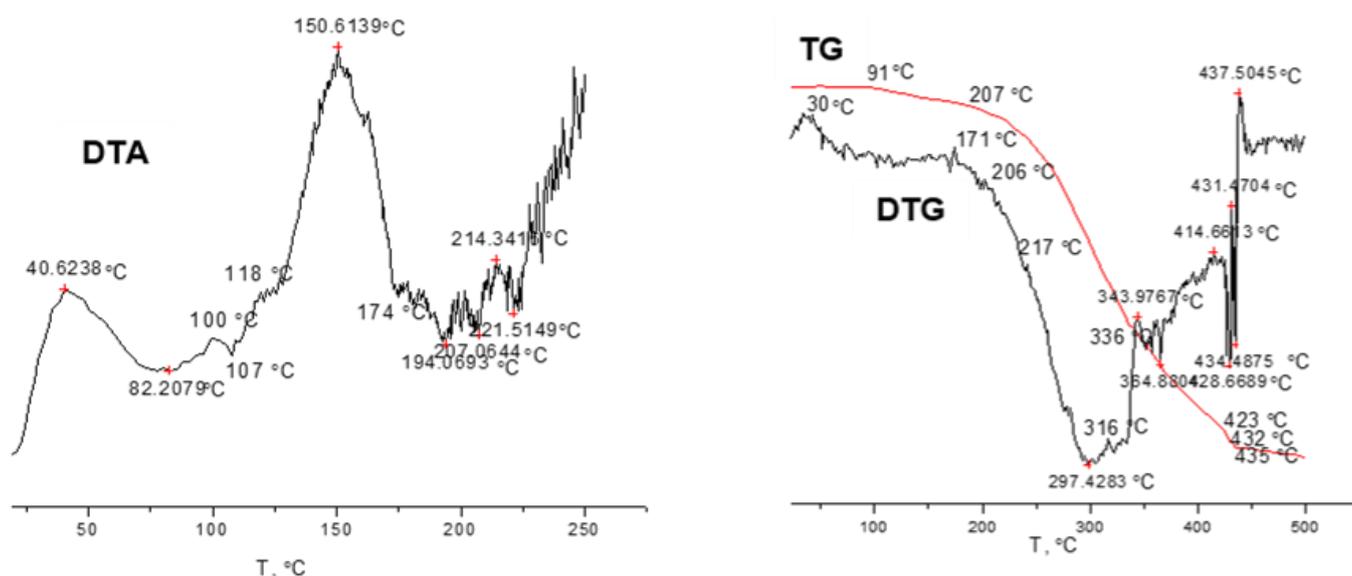


Fig. 2. DTA, TG, and DTG curves of PS-derived FAA.

Table 3. Composition and technical properties of emulsions prepared with FAA

Composition of emulsions				Technical properties of emulsions			
Surfactant-product, wt%	Aqueous phase, wt%		Diesel oil, wt%	Viscosity, Pa·s	Thermal stability at 80 °C, days	Stability to phase separation, days	Electrical stability, V
	H ₂ O	CaCl ₂					
PS _s (MEA) – 0.1	40	7	52.9	0.150	0	1	60
PS _s (MEA) – 2.5	40	7	50.5	0.215	>9	>90	220
PS _s (HED) – 2.5	40	7	50.5	0.189	8	>90	170
PS _s (BHED) – 2.5	40	7	50.5	0.205	>9	>90	200
PS _r (MEA) – 2.5	40	7	50.5	0.192	>9	>90	180
PS _r (HED) – 2.5	40	7	50.5	0.214	7	>90	190
PS _r (BHED) – 2.5	40	7	50.5	0.179	>9	>90	150

PS_s and PS_r = phosphatidic sludge derived from sunflower and rapeseed oils, respectively. MEA = monoethanolamine, HED = *N*-(2-hydroxyethyl)ethylenediamine. BHED = *N,N'*-bis(2-hydroxyethyl)ethylenediamine.

Conclusions

This work explored the potential of phosphatidic sludge as a promising non-food-competitive feedstock for the production of technical surfactants. It employs undervalued phosphatides derived from sunflower and rapeseed oils to the base-catalysed amidation with monoethanolamine, *N*-(2-hydroxyethyl)ethylenediamine, or *N,N'*-bis(2-hydroxyethyl)ethylenediamine. The study demonstrated that the catalyst, calcium hydroxide, allows high yields (95–98 %) of targeted fatty acid alkanolamides, and simultaneously coordinates glycerolphosphatides, which render their high solubility in non-polar organic media. At the same time, thermal analyses of the synthesised products ensure their

stability at elevated temperatures (up to 180 °C). These altogether led to the exploitation of biobased surfactants as emulsifiers for the creation of highly stable reversed emulsions for potential application as drilling fluids are recommended for the disclosure of productive strata; perforation of wells and development of productive layers; blockage of gas, gas condensate and oil wells; elimination of manifestations and flow of gas in wells; limitation and elimination of waterways; cleaning of the hollow zone of wells and intensification of the inflow of hydrocarbon raw materials, which have been tested in laboratory and experimental industrial conditions, and a significant part of them have been introduced or tested on gas condensate fields.

Creation of coordinated, effective and economical actions that should be formed in the state energy policy of Ukraine would facilitate the development of oil and gas companies, namely: increase of own oil and gas production; maximizing the potential of energy saving; diversification of external sources of supply; approximation of the parameters of the oil and gas industry to the norms and standards of the European Union.

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Побічні продукти виробництва рослинних олій як сировина для створення поверхнево-активних речовин та технічних систем

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Виходячи з оцінки технічного аналізу, поточного виробництва й споживання рослинних олій та побічних продуктів від їх виробництва, визначено потенційну сировину для синтезу ПАР, що не конкурує з харчовою продукцією. Це фосфатиди (фосфатидні концентрати) низької вартості, які можливо безпосередньо використовувати для хімічної трансформації. Створено поверхнево-активні речовини на біологічній основі трансамідуванням фосфатидного концентрату, отриманого при рафінуванні соняшникової та ріпакової олій, моноетаноламіном, N-(2-гідроксіетил)етилендіаміном або N,N'-біс(2-гідроксіетил)етилендіаміном під дією кальцію гідроксид як реагента каталізатора з ефективними виходами (95-98 %). Окрім усунення відходів, використання фосфатидів дозволило розробити композиції з використанням ПАР, які включають алкілоламіди жирних кислот та гліцеролфосфатиди кальцію з покращеною розчинністю в органічних неполярних розчинниках. З розробленими поверхнево-активними речовинами були створені інвертні емульсійні системи, які можуть застосовуватись для розробки та експлуатації родовищ нафти та газу: для буріння свердловин, розкриття продуктивних пластів; перфорації свердловин, освоєння продуктивних пластів; глушіння газових, газоконденсатних і нафтових свердловин; усунення проявів і плинину газу в свердловинах; обмеження та ліквідації водопроявів; очищення привибійної зони свердловин, інтенсифікації припливу вуглеводневої сировини. Інвертні емульсії випробувані в лабораторних і дослідно-промислових умовах, значна частина з них впроваджена чи апробована на газоконденсатних родовищах. Скоординовані, ефективні та економічні дії, які мають сформуватись у державній енергетичній політиці України, сприятимуть розвитку нафтогазодобувних підприємств, а саме: збільшенню видобутку власних нафти і газу; максимальному залученню потенціалу енергозбереження; диверсифікації зовнішніх джерел постачання; наближенню параметрів нафтогазодобувної галузі до норм і стандартів Європейського Союзу.

Ключові слова: фосфатидний концентрат, органічний синтез, поверхнево-активні речовини, інвертні емульсії.