

ABSTRACT AND REFERENCES

TECHNOLOGY ORGANIC AND INORGANIC SUBSTANCES

DOI: 10.15587/1729-4061.2018.123649

ELECTROCHEMICAL REGENERATION OF OXYGEN-CONTAINING COMPOUNDS IN THE EXTRACTS OF USED OILS (p. 4-9)

Oleksandr Davydenko

National Aviation University, Kyiv, Ukraine

ORCID: <http://orcid.org/0000-0002-4502-7931>

Volodymyr Ledovskykh

National Aviation University, Kyiv, Ukraine

ORCID: <http://orcid.org/0000-0002-8613-889X>

Growth in the volumes of oils applied for various purposes, including motor oils, results in the formation of large quantities of toxic waste – used oils. At the same time, they are a valuable raw material for the production of fresh oils by their regeneration though existing techniques have a number of shortcomings, the main of which is the formation of new waste that is difficult to dispose of.

It is shown that the extraction of used oils by an alkaline aqueous-alcohol solution enables an almost complete removal of oxygen-containing compounds, as evidenced by the reduced acid number, from 1.76 mg KOH/g of oil to 0 mg KOH/g of oil. This results in a significant decrease in corrosion aggressiveness of the medium.

The results of research into electrochemical processes in model solutions of ketones, aldehydes and carboxylic acids revealed the possibility to transform their products on electrodes into non-toxic useful products – carbohydrate products.

The results of research into electrochemical processes in model solutions of oxygen-containing compounds and extracts of used oils at anodic potentials of 2.4 V demonstrated the possibility of their transformation on electrodes into useful carbohydrate products. This would contribute to an increase in the output of regenerated oil, prevents the formation of toxic waste.

**Keywords:** carboxylic acid electro-oxidation, electroreduction of aldehydes and ketones, regeneration, used oil.

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DOI: 10.15587/1729-4061.2018.122938

RESEARCH INTO TRANSESTERIFICATION OF TRIGLYCERIDES BY ALIPHATIC ALCOHOLS C<sub>2</sub>–C<sub>4</sub> IN THE PRESENCE OF IONITES (p. 10-16)

Yuriy Melnyk

Lviv Polytechnic National University, Lviv, Ukraine

ORCID: <http://orcid.org/0000-0003-0109-5526>

**Stepan Melnyk**

Lviv Polytechnic National University, Lviv, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-0629-9723>

**Zoryana Palyukh**

Lviv Polytechnic National University, Lviv, Ukraine  
**ORCID:** <http://orcid.org/0000-0003-1155-7763>

**Bohdan Dzinyak**

Lviv Polytechnic National University, Lviv, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-1824-2871>

We studied the reaction of transesterification of triglycerides of sunflower oil by aliphatic alcohols C<sub>2</sub>–C<sub>4</sub> in the presence of the cationite CU-2-8 in H-form and with the immobilized ions of metals and the anionite AV-17-8. We investigated the influence of the type of ionite, immobilized cation of metal, the structure of alcohol, molar ratio of triglyceride:alcohol, temperature, and the content of catalyst in the reaction mixture on the rate of reaction of transesterification and conversion of triglycerides. It was established that the activity of catalysts, based on the cationite CU-2-8 with immobilized ions of metals, in the reaction of transesterification of triglycerides by alcohols C<sub>2</sub>–C<sub>4</sub> depends on the type of the immobilized ion of metal. It was shown that the cationite CU-2-8 with immobilized ions of Ni<sup>2+</sup>, Sn<sup>2+</sup>, Co<sup>2+</sup> and in H-form demonstrates the highest activity in the reaction of transesterification. It was established that in the presence of the examined catalysts conversion of triglycerides decreases with an increase in the length of the alcohol chain. Extreme dependence of the rate of transesterification reaction and conversion of triglycerides on the content of the catalyst-ionite was established. Optimum conditions for the reaction of transesterification of triglyceride by aliphatic alcohols C<sub>2</sub>–C<sub>4</sub> were found. It was established that optimal content of catalyst is 2 % by weight; molar ratio of alcohol:TG for ethanol, propane-1-ol and propane-2-ol is (4–5):1, for butane-1-ol, it is 10:1.

The temperature of reaction should be close to the boiling temperature of alcohol. It was shown that the examined catalysts demonstrate high activity in the reaction of transesterification of triglycerides and make it possible to achieve high conversion rate of starting materials. The results obtained indicate the feasibility of applying catalysts based on the cationite CU-2-8 with immobilized ions of metals in the reaction of transesterification of triglycerides by alcohols C<sub>2</sub>–C<sub>4</sub>.

**Keywords:** transesterification, aliphatic alcohols C<sub>2</sub>–C<sub>4</sub>, triglycerides, ionite, ions of metals.

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**DOI:** [10.15587/1729-4061.2018.123885](https://doi.org/10.15587/1729-4061.2018.123885)

**EFFECT OF MECHANOACTIVATED CHEMICAL ADDITIVES ON THE PROCESS OF GAS HYDRATE FORMATION (p. 17-26)**

**Volodymyr Bondarenko**

National Mining University, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-7552-0236>

**Olena Svietkina**

National Mining University, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0003-0857-8037>

**Kateryna Sai**

National Mining University, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0003-1488-3230>

This study addresses the production of gas hydrate of methane with a high gas-hydrate-forming content in a solid phase in the isolated system at T=274 K and pressure of 5 MPa and presence of mechanically activated rocks close to the bottom of the chamber.

We used mechanically activated samples of various degrees of grinding to increase an area of contact surface of heterogeneous phases. We carried out mechanochemical activation of materials in a vertical vibrating mill (VVM).

In the study, we found out that formation of gas hydrates on activated aluminosilicates leads to the cryochemical synthesis of hydrocarbons, due to formation of additional reaction centers formed upon activation. This indicates a change in the mechanism of formation of GH during the process. We calculated three rate constants for the formation of GH of methane, which vary from  $1.20 \cdot 10^{-2}$  to  $1.25 \cdot 10^{-2}$  hour<sup>-1</sup>, based on semi-logarithmic anamorphosis. The study showed that formation of methane gas hydrates in presence of activated additives leads to formation of up to 5–6 % of ethane. Chromatographic method confirmed this.

This indicates possibility of carrying out a low-temperature synthesis of higher hydrocarbons in the artificial production of GH, in contrast to the known mechanochemical transformations during the process of obtaining gas from gas hydrates.

**Keywords:** methane gas hydrates, mechanoactivation, heterogeneous catalysis, rate of hydrate formation, dissociation, aluminosilicates, phase transformations.

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**DOI:** 10.15587/1729-4061.2018.121595

**DEFINITION OF SYNTHESIS PARAMETERS  
OF ULTRAFINE NICKEL POWDER BY DIRECT  
ELECTROLYSIS FOR APPLICATION IN SUPERALLOY  
PRODUCTION (p. 27-33)**

**Vadym Kovalenko**

Ukrainian State University of Chemical Technology, Dnipro,  
Ukraine

Vyatka State University, Kirov, Russian Federation  
**ORCID:** <http://orcid.org/0000-0002-8012-6732>

**Valerii Kotok**

Ukrainian State University of Chemical Technology, Dnipro,  
Ukraine

Vyatka State University, Kirov, Russian Federation  
**ORCID:** <http://orcid.org/0000-0001-8879-7189>

**Vlasov Sergey**

National Mining University, Dnipro, Ukraine  
Vyatka State University, Kirov, Russian Federation  
**ORCID:** <http://orcid.org/0000-0002-5537-6342>

The optimization of the synthesis method of ultrafine nickel powder for the production of superalloys, by means of direct electrolysis of the nickel ammine complex, has been conducted. The influence of electrolyte temperature and ammonia concentration on the electrolysis process and powder characteristics has been studied. It has been revealed that an increase in electrolyte temperature leads to a larger particle size of the powder and formation of compact metal particles. It has been determined that the maximum electrolyte temperature must not exceed 50 °C. By recording the polarization curves, it was established that an increase in ammonia concentration leads to increased polarization of nickel formation and formation of finer powder. Lowering of ammonia concentration leads to contamination of the powder with barely soluble hydroxyl compounds of nickel. Analysis of the anodic curves has revealed that the nickel anode is partly soluble. It has been discovered that the addition of Trilon B to the electrolyte has a positive effect on powder characteristics. The powder formed under optimal conditions was composed of coral-like particles with the size of 40–70 µm, which could be easily ground to their spherical components if no compact metal particles were present. High purity of the powder was confirmed by the EDX. The cathodic and anodic current yields were determined to be: 35–41 % and 5–8 % respectively.

**Keywords:** nickel ammine complex, nickel powder, superalloys, dendrite, Trilon B.

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**DOI:** 10.15587/1729-4061.2018.119624

**A COMPARATIVE STUDY ON THE INFLUENCE OF METAKAOLIN AND KAOLIN ADDITIVES ON PROPERTIES AND STRUCTURE OF THE ALKALI ACTIVATED SLAG CEMENT AND CONCRETE (p. 33-39)**

**Pavel Krivenko**

Scientific Research Institute for Binders and Materials, Kyiv National University of Construction and Architecture, Kyiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-7697-2437>

**Oleg Petropavlovskyi**

Scientific Research Institute for Binders and Materials, Kyiv National University of Construction and Architecture, Kyiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-3381-1411>

**Oleksandr Kovalchuk**

Scientific Research Institute for Binders and Materials, Kyiv National University of Construction and Architecture, Kyiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-6337-0488>

The influence of the metakaolin and kaolin additives on the formation and properties of the alkali-activated slag cements and concretes was studied.

The influence of the metakaolin and kaolin additives on macro- and microstructure formation of the cements and concretes was studied.

A conclusion was drawn that the processes of microstructure formation of the cement stone with the additive flow in a similar sequence but with different intensity. A conclusion was drawn that the addition of the kaolin instead of metakaolin affected as follows: 2.5–10 % by mass reduced the value of NCP by 9.5–8.7 %, respectively; 2.5–5 % by mass did not affect setting times, but with increase up to 10 % by mass the initial setting time was shorter (from 48 min to 40 min); 2.5–5 % by mass did not affect compressive strength at all stages of hardening, but with increase up to 10 % by mass reduced strength characteristics of the cement-sand specimens (from 57.0 MPa to 49.0 MPa).

In case of the addition of 5 % kaolin by mass, an optimal macrostructure of the concrete is formed in which the quantities of the "conditionally" closed pores are by 17.7 % higher compared to those of the concretes with the same quantities of the metakaolin. This resulted in the higher freeze/thaw resistance of the concrete (from F400 up to F500). Based on the comparison of properties and structure of the cement and concrete containing the kaolin and metakaolin additives, a possibility to substitute metakaolin by kaolin as a correcting additive was established.

**Keywords:** alkali activated cement, compressive strength, concrete, kaolin, metakaolin, freeze/thaw resistance.

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DOI: 10.15587/1729-4061.2018.124085

## RESEARCH AND CONTROL OF THE PURITY OF PRODUCTION HYDROGEN WITH A HIGH DEGREE OF PURIFICATION WHEN APPLYING THE ELECTROLYSIS METHOD OF PRODUCTION (p. 40-46)

Valeriy Nikolsky

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine

ORCID: <http://orcid.org/0000-0001-6069-169X>

Olga Oliynyk

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine

ORCID: <http://orcid.org/0000-0003-2666-3825>

**Viktor Ved**

Ukrainian State University of Chemical Technology, Dnipro,  
Ukraine

**ORCID:** <http://orcid.org/0000-0002-2391-6463>

**Olena Gnatko**

Ukrainian State University of Chemical Technology, Dnipro,  
Ukraine

**ORCID:** <http://orcid.org/0000-0003-4376-3860>

**Andrii Pugach**

Dnipro State Agrarian and Economic University, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-5586-424X>

**Iuliia Bartashevská**

Alfred Nobel University, Dnipro, Ukraine

**ORCID:** <http://orcid.org/0000-0002-0300-0693>

We conducted experimental studies aimed at determining the purity of hydrogen obtained at the electrolysis installation made by Hydrogen Technologies (Norway) at the pipe plant Centravis Production Ukraine in the city of Nikopol, Ukraine.

It was established that the determination of hydrogen purity and the degree of its purification from impurities (nitrogen) in microconcentrations involves two stages of measurements:

- research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of  $[[\text{N}_2]] 0.001\text{--}0.01$  % (rough estimate);

- research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of  $[[\text{N}_2]] 0.001\text{--}0.01$  % (fine assessment).

We determined that the purity of production hydrogen, obtained during research, was  $99.9\pm0.1$  %. A given value for purity does not match certification indicators for purity of production hydrogen claimed by the manufacturer to equal 99.9999 %.

We analyzed the reasons for the mismatch between the purity of obtained hydrogen and claimed characteristics. A detailed analysis revealed that the possible cause of high nitrogen concentration in hydrogen is the worn piston rings in the stage of compressor pistons, which causes the penetration of nitrogen in microconcentrations into production hydrogen. Piston rings in the compressor's stage were replaced. Repeated studies into purity of production hydrogen indicate that the purity of production hydrogen amounted to  $99.99\pm0.01$  %, which corresponds to the hydrogen of grade A.

**Keywords:** electrolysis installation, degree of purification, concentration of technical hydrogen, impurity, chromatography, digital filtering.

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DOI: 10.15587/1729-4061.2018.123852

**STUDY OF ANODE PROCESSES DURING DEVELOPMENT OF THE NEW COMPLEX THIOCARBAMIDE-CITRATE COPPER PLATING ELECTROLYTE (p. 47-51)**

**Olha Smirnova**

National Technical University  
«Kharkiv Polytechnic Institute», Kharkiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-9869-7007>

**Alexei Pilipenko**

National Technical University  
«Kharkiv Polytechnic Institute», Kharkiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-5004-3680>

**Hanna Pancheva**

National Technical University  
«Kharkiv Polytechnic Institute», Kharkiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-9397-3546>

**Alexei Zhelavskyi**

V. N. Karazin Kharkiv National University, Kharkiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-9240-8446>

**Kateryna Rutkovska**

National Technical University  
«Kharkiv Polytechnic Institute», Kharkiv, Ukraine  
**ORCID:** <http://orcid.org/0000-0003-0460-1906>

The kinetics of anodic reactions occurring on copper in thiocarbamide-citrate solutions was studied. Thiocarbamide forms stable copper (I) complexes of the cationic type with a coordination number equal to unity. Citric acid ensures acid pH value of electrolyte and causes active dissolution of copper under conditions of anode polarization. The joint presence of  $\text{CS}(\text{NH}_2)_2$  and  $\text{C}_6\text{H}_8\text{O}_7$  in the solution contributes to the copper electrode activation under conditions of anodic polarization. Increasing the concentration of thiocarbamide leads to a drastic shift of copper dissolution potentials towards the region of negative values. Study of the kinetics of anodic behavior of copper by acquiring the voltammograms revealed the nature of the limiting stage of reaction.

It is shown that the process of dissolution in a thiocarbamide-citrate electrolyte is controlled by the diffusion phase. This is confirmed by the results of graphical processing of polarization dependences in coordinates  $\eta - \lg(1 - j_a/j_d)$ . An increase in  $v_p$  within  $5-100 \text{ mV}\cdot\text{s}^{-1}$  causes an increase in  $j_d$  from  $2.2$  to  $12.0 \text{ mA}\cdot\text{cm}^{-2}$ , which indicates diffusion control over the process. The process of copper dissolution proceeds under stationary mode with uniform etching of intragrain boundaries and volume of the metal's grain.

**Keywords:** complex compounds, anodic polarization, limiting stage, diffusion overvoltage, polarization dependence.

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DOI: 10.15587/1729-4061.2018.123896

**THE STUDY OF PROPERTIES OF COMPOSITE ADSORPTIVE MATERIALS “SILICA GEL – CRYSTALLINE HYDRATE” FOR HEAT STORAGE DEVICES (p. 52-58)**

**Kostyantyn Sukhyj**

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-4585-8268>

**Elena Belyanovskaya**

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0003-1873-4574>

**Vadym Kovalenko**

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine  
Vyatka State University, Kirov, Russian Federation  
**ORCID:** <http://orcid.org/0000-0002-8012-6732>

**Valerii Kotok**

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine  
Vyatka State University, Kirov, Russian Federation  
**ORCID:** <http://orcid.org/0000-0001-8879-7189>

**Mikhaylo Sukhyj**

Ukrainian State University of Chemical Technology,  
Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-3906-4592>

**Olena Kolomiyets**

National metallurgical academy of Ukraine, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0002-3216-649X>

**Mykhailo Gubynskyi**

National metallurgical academy of Ukraine, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-5061-7779>

**Oleksandr Yeromin**

National metallurgical academy of Ukraine, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-8306-578X>

**Olena Prokopenko**

National metallurgical academy of Ukraine, Dnipro, Ukraine  
**ORCID:** <http://orcid.org/0000-0001-6151-0301>

Heat energy storage is one of the most common technical solutions in the conditions of operation of low-potential and renewable energy sources. Adsorption heat energy storage devices based on the composite media “silica gel – salt” are the most effective in these conditions. The technique and technology of sol-gel synthesis of the composite adsorption materials “silica gel – sodium sulfate” and “silica gel – sodium acetate” have been developed. A special feature of this technique is a two-stage process involving the formation of silicon phase nuclei in the interaction of aqueous solutions of silicate glass and sulphuric or acetic acids in the presence of a polymeric quaternary ammonium salt and subsequent coarsening of the particles with the gradual addition of solutions of silicate glass and the corresponding acids. The essence of the technology consists in successive stages of formation and integration of the silicic phase nuclei, hydrolysis of functional OH- groups, filtration and drying of the fine precipitate. A qualitative difference in the adsorption properties of the synthesized composites and the mechanical mixture of salt – silica gel with sorption capacity inferior to them on average by 30 % is revealed by differential thermal analysis. The processes of application of the composite adsorption materials “silica gel – sodium sulfate” and “silica gel – sodium acetate” obtained by the sol-gel method have been studied. A qualitative difference in the kinetics of adsorption of water by the composite adsorbents is shown as compared to massive salts. It is established that the amount of heat of adsorption of water vapor by the composite adsorbents of the materials “silica gel – sodium sulfate” and “silica gel – sodium acetate” is approximately 30 % greater than the linear superposition of salt and silica gel.

**Keywords:** heat-accumulating materials, composite sorbents, sol-gel synthesis, adsorption heat, energy storage density, kinetics of hydration.

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