Plasma-Assisted Reforming of Ethanol in Dynamic Plasma-Liquid System: Experiments and Modeling

Valeriy Ya. Chernyak, Sergej V. Olszewski, Vitalij V. Yukhymenko, Elena V. Solomenko, Iryna V. Prysiazhnevych, Vadym V. Naumov, Dmitry S. Levko, Anatolij I. Shchedrin, Andriy V. Ryabtsev, Valentina P. Demchina, Vladimir S. Kudryavtsev, Eugene V. Martysh, and Maxim A. Verovchuck

***Abstract*—This paper presents the results of experimental and theoretical investigations of the process of nonthermal plasma-assisted reforming of aqueous ethanol solutions in the dynamic plasma-liquid system based on the electric discharge in a gas channel with liquid wall. The experiments show possibilities and efficiency of low-temperature plasma-chemical conversion of liquid ethanol into synthesis gas. The numerical modeling clarifies the nature and explains the kinetic mechanism of nonequilibrium plasma-chemical transformations in the system.**

***Index Terms*—Electric discharge, nonequilibrium plasma chemistry, nonthermal plasma, plasma-assisted fuel reforming.**

I. Introduction

TODAY, a plasma-assisted fuel reforming is of great scientific and practical interest [1]–[3]. The conversion of heavy hydrocarbons (HC) into the free hydrogen (H_2), carbon monoxide (CO), and other easily burning fractions allows the improvement of the efficiency of combustion and the reduction of atmospheric air pollution [4]. Research on alternative biofuels which can replace traditional fossil fuels, petrol, and natural gas is also very actual due to environmental and energy saving problems [5]. Thus, bioethanol (ethyl alcohol-based fuel produced from agricultural biomass) is already used as a motor fuel in spark-ignition gasoline engines. However, the quality of this fuel is still an open question. Ethanol has a number of limitations including a relatively low specific heat of combustion and poor storage because of high volatility and water absorption. Therefore, it is timely to research possible ways of the plasma reforming of biofuels enhancing their combustion efficiency.

There are various methods of the plasma-assisted HC fuel conversion using thermal and nonthermal plasma sources [6]. Thermal plasma, which is in thermodynamic equilibrium, has characteristics of high ionization and higher energetic density. This has merits of fast decomposition of hydrocarbons but demerits of poor selectivity of chemical transformations and high expenditure of electric power ($>$ 1 kW). Nonthermal plasma, which is in kinetic nonequilibrium, has characteristics of low ionization but benefits of high reactivity and selectivity of plasma-chemical transformations, providing high-enough productivity at relatively low power consumption ($<$ 1 kW). This can be obtained by a high voltage discharging in the flow at atmospheric pressure [6].

Applying the nonequilibrium reacting plasma to the fuel reforming, different techniques were tested worldwide, e.g., radio-frequency discharge [7], microwave discharge [8], corona discharge [9], spark discharge [10], dielectric barrier discharge [11], pulsed discharge [12], gliding arc discharge [13], transient gliding arc in tornado [14], and various hybrid discharges. In fact, all systems have their merits and demerits depending on the use; therefore, it is hard to compare [15]. According to recent reports from the Massachusetts Institute of Technology [16] where nonthermal low-current plasmatrons were used for the reforming of real fuels, gasoline, diesel, and biofuels for motor vehicles, the effect of plasma reforming on vehicles' motor operations was always positive: The motor performance had grown up, whereas NO_x/soot exhaust had reduced; at that, electrical energy consumption did not exceed few percents of the hydrogen caloric capacity.

Among possible types of electric discharges, which can produce nonthermal plasma, two specific cases are of current interest in Kiev National University [17]–[26]. One is a dynamic plasma-liquid system (PLS) based on a transverse arc in a blowing flow (BTA) [18]. The BTA is an intermediate case of a self-sustained high-voltage discharge with the arc adjusted by a transverse gas flow. It differs from the nonstationary gliding arc of Czernichowski type by a fixed arc length. It also has a convective cooling of the plasma column by a gas flow but without heat losses at the walls because it is a free arc jet. An intensive transverse ventilation of BTA plasma increases its ionization nonequilibrium and nonisothermality that is of fundamental importance for plasma chemistry. While the most of discharge energy goes into the mean energy of electrons and not just into thermal heating, it gives a desirable reactivity and selectivity of plasma chemical transformations. The BTA was tested successfully in the different PLS as a primary and secondary discharge for plasma processing of liquid HC substances (acetone, toluene, etc.) including both plasma-enhanced decomposition and plasma-assisted combustion [19]–[21].

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V. Ya. Chernyak, S. V. Olszewski, V. V. Yukhymenko, E. V. Solomenko, I. V. Prysiazhnevych, V. V. Naumov, E. V. Martysh, and M. A. Verovchuck are with the Faculty of Radiophysics, Kyiv National Taras Shevchenko University, 03122 Kyiv, Ukraine (e-mail: chern@univ.kiev.ua; chernyak_v@ukr.net; solaristics@gmail.com; vitaliy@ukr.net; andin@univ.kiev.ua; priv@ukr.net; emart@univ.kiev.ua; naumov_vv@ukr.net).

D. S. Levko, A. I. Shchedrin, and A. V. Ryabtsev are with the Institute of Physics, National Academy of Sciences of Ukraine, 03028 Kyiv, Ukraine (e-mail: ashched@iop.kiev.ua).

V. P. Demchina and V. S. Kudryavtsev are with the Gas Institute, National Academy of Sciences of Ukraine, 03028 Kyiv, Ukraine.

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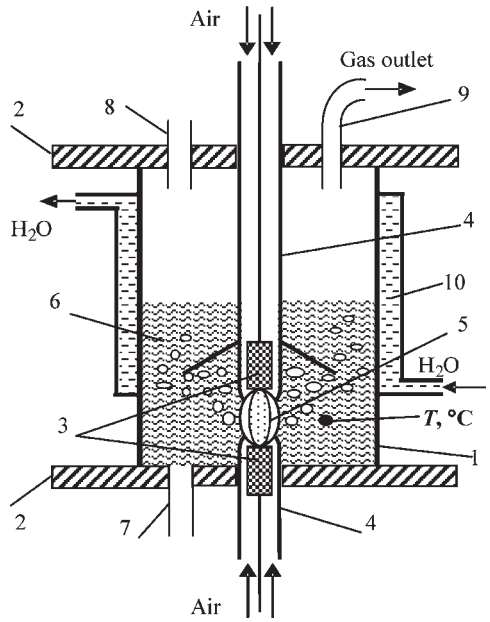

Fig. 1. Schematic of a PLS reactor with a DGCLW.

Another source of nonthermal plasma, which can provide simultaneously a high level of nonequilibrium and high density of reacting species in the PLS, is an electric discharge in a gas channel with liquid wall (DGCLW). Its main features are the following: 1) well-developed plasma-liquid interface and large surface-to-volume ratio; 2) wide possibilities of control of plasma-created gas and liquid-phase components during plasma processing; 3) opportunity of using nonclassic liquids including colloidal solutions and mash; 4) capability of both dc and ac operating modes (in contrast to diaphragm or capillary discharges). The DGCLW was realized initially in plasma processing of HC organics (phenol) in air-water systems [22]. Then, the DGCLW was utilized for fuel reforming in ethanol–water mixtures [23]. The main idea is that the DGCLW can be burning directly within liquid fuel without preliminary gasification. Really, the DGCLW has already demonstrated its ability in conversion of ethanol into the hydrogen-enriched synthesis gas following by enhanced combustion [24], [25]. However, it needs comprehensive investigations.

In this paper, we report new results of our experimental and theoretical studies of the process of plasma-assisted reforming of ethanol in the PLS with the DGCLW using available methods of diagnostics and numerical modeling.

II. Experiments and Results of Measurements

A. Experimental Details

Experiments were done with a PLS reactor of the DGCLW type whose schematic is shown in Figs. 1 and 2. It consists of a cylindrical quartz test vessel (1) sealed at the top and bottom by duralumin flanges (2) with a built-in electrode system (3). The cooper rod electrodes (3) were inserted into the quarts tubes (4) and installed coaxially one opposite the other. The tubes (4) served also for the gas (air) inlet. A compressed atmospheric air was injected along electrodes (3) through the open nozzle

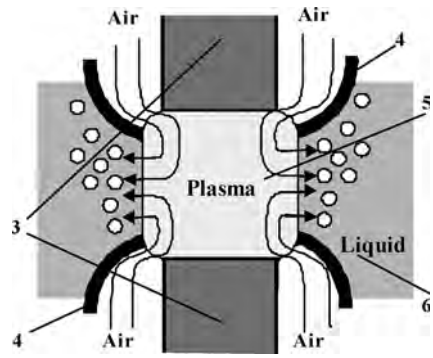

Fig. 2. Schematic of the DGCLW.

ends (4) and formed a stable counter-flow gas channel (5) surrounded by liquid ethanol (6). The electric discharge was burned in the gas channel between the immersed electrodes where an electric breakdown occurred. The ethanol solution was filled into the reactor through the drain pipe (7) at the bottom flange. The outlet connections (8) and (9) at the top flange were connected with a system of communicating vessels allowing control of the liquid level and pressure in the reactor. The outlet pipe (9) served for the transportation of the synthesis gas products from the reactor to the condensing vessel and further to the gas analysis. Because of the electric-discharge heat release and heating of plasma-treated solution in the reactor, the auxiliary cooling was provided by the water-cooled jacket (10). The temperature of work solution in the reactor was measured by the immersed thermocouple.

The test solutions of ethanol were prepared by diluting alcohol (C_2H_5OH) in distilled water (H_2O). A standard volume of treated liquid was 100 cm^3 at the reactor capacity of 200 cm^3. The ethanol rate in the reactor was controlled by a visual change of the liquid level in the communicating volumetric vessel. The input air flow rate was controlled by the rotameter. The output gas flow rate was controlled by the rate of the water drive from the calibrated volume (1.08 L). After condensation of residual ethanol vapors, the synthesis gas products were collected in the special glass containers (0.5 L).

The ethanol solutions were processed in the reactor under different discharge modes with the constant airflow G and without airflow, when the input airflow was stopped after the discharge initiation.

The discharge worked in the continuous regime powered by the dc supply with a ballast resistor. The typical discharge voltages are $U = 1.5$–1.2 kV, and discharge currents are $I = 100$–200 mA. The gap between electrodes, gas flow rates, ethanol–water mixing ratio, and discharge processing time were varied in order to optimize the process.

The discharge plasma in the reactor was diagnosed by the UV-NIR optical emission spectroscopy (OES) using a high-speed PC-operated multichannel CCD-based diffraction spectrometer Solar SL40-2-3648 (range of wavelengths $\lambda = 200$–1100 nm, spectral resolution FWHM $\sim$0.65 nm).

The component content of the output synthesis gas products after the reactor was analyzed by the mass spectrometry using a monopole mass spectrometer MX 7301 and by the gas-phase

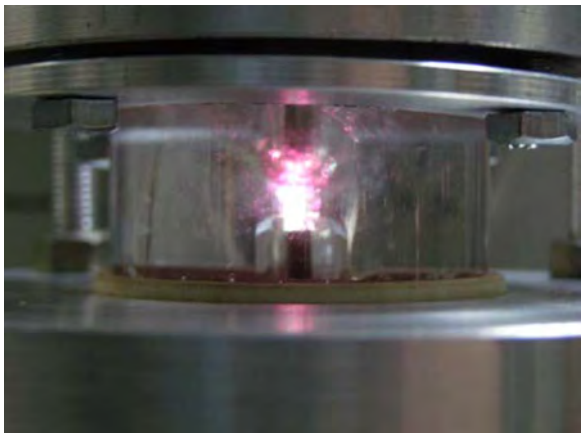

Fig. 3. DGCLW in a flow of air in water.

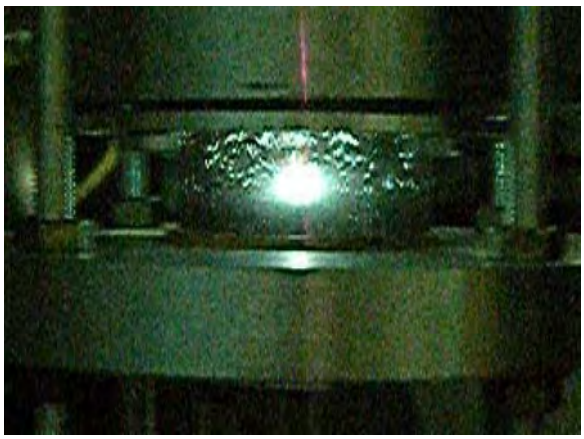

Fig. 4. DGCLW in a flow of air in ethanol.

chromatography using a gas chromatograph 6890 N Agilent with the calibrated thermal conductivity detectors.

B. Experimental Results

The photographs of the DGCLW working in the PLS reactor with water and with ethanol are shown in Figs. 3 and 4, respectively.

The typical current-voltage characteristics of the DGCLW in the 50% ethanol–water solution are shown in Fig. 5. Here, the electrode gap is 2 mm. A dropping character of I–V curves at currents from 100 to 400 mA indicates the transition regime from the abnormal glow to the arc discharge.

The typical optical emission spectrum from the DGCLW during the ethanol processing is shown in Fig. 6. It is multicomponent and contains molecular bands of OH UV system ($A^2\Sigma^+ - X^2\Pi$: (0–0) 306.4–308.9 nm), N_2 2^+ system ($C^3\Pi_u - B^3\Pi_g$: (0–0) 337.1, (0–1) 357.7, (0–2) 380.5, (1–4) 399.8 nm, etc.), CN ($B^2\Sigma^+ - X^2\Sigma^+$: (0–0) 388.3 nm), C_2 Swan band ($d^3\Pi_g - a^3\Pi_u$: (0–0) 516.5, (1–1) 512.9 nm, etc.), atomic lines of H Balmer series (656.3, 486.1 nm), OI (777.1, 844.6, 926.6 nm), CuI (324.7, 327.4, 465.1, 510.5, 515.3, 521.8 nm), and other emissions which identify a nonequilibrium nature of the DGCLW plasma. The H emission indicates the vaporization and dissociation of ethanol and water molecules and production of hydrogen, the C_2 emission relates to the carbon formation,

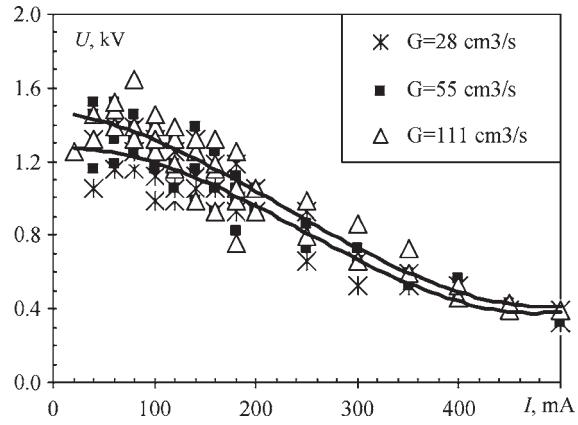

Fig. 5. Current–voltage characteristics of the DGCLW in the 50% ethanol–water solution at different airflow rates G.

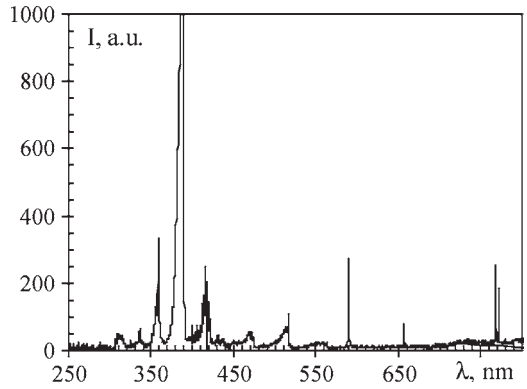

Fig. 6. Optical emission spectrum from the DGCLW plasma during the ethanol processing.

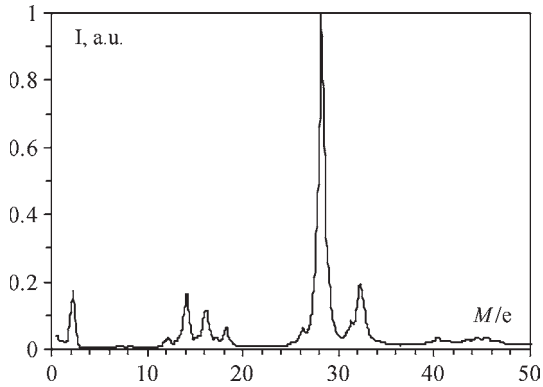

Fig. 7. Mass spectrum of output gas products after the ethanol processing in the DGCLW.

and the N_2 and CN emissions relate to the dissociating air [26]. The OES simulation of selected spectral lines (Cu, O, H) and bands (N_2) gives the character electronic and vibration temperatures in the range of 0.6 (T_e) and 0.35 eV (T_v).

The typical mass spectrum of output gas products registered after the ethanol processing in the PLS-DGCLW reactor is shown in Fig. 7. It contains the gas-phase components related to the mass ratios M/e = 2(H_2^+), 12 (C^+), 14 (N^+), 16 (O^+, CH_4^+, 18 (H_2O^+), 28 (CO^+, N_2^+), and 32 (O_2^+).

The quantitative results of the measurements of concentrations of basic components in output gas products after the

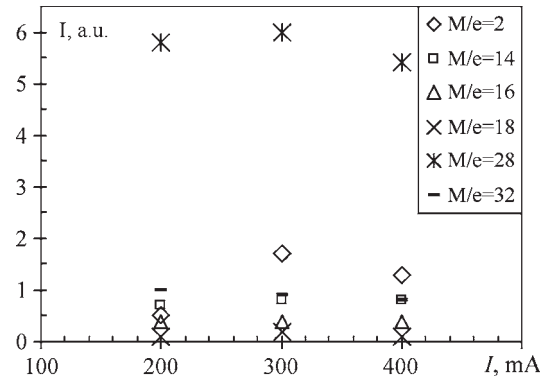

Fig. 8. Mass-spectrometry data on the composition of output gas products after the ethanol processing in the DGCLW.

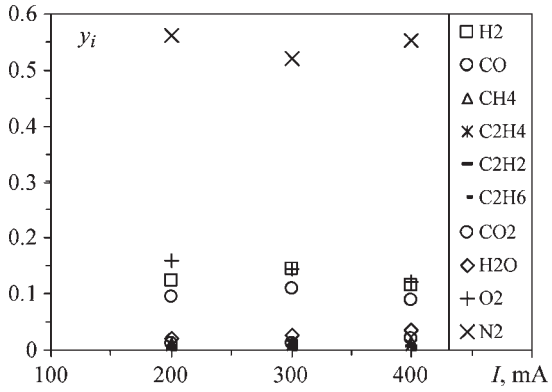

Fig. 9. Gas-chromatography data on the composition of output gas products after the ethanol processing in the DGCLW.

ethanol processing at different discharge currents $I = 200$, 300, and 400 mA are shown in Fig. 8 (mass spectrometry) and Fig. 9 (gas chromatography). The data are given for the case of mixture $C_2H_5OH : H_2O = 1 : 1$ and air flow rate $G = 55$ cm^3/s.

One can see that results of gas chromatography and mass spectrometry correlate well and that H_2 and CO are the main components of synthesis gas (syngas) produced from ethanol in the DGCLW reactor. The fractional amount of H_2 and CO in the syngas reaches ~87%–89%, i.e., many times higher than for all hydrocarbons including CH_4, C_2H_2, C_2H_4, and C_2H_6 (the total syngas amount in the output gas products is ~30% by volume). It is difficult to set a common tendency because different components have ambiguous behavior. However, with increasing discharge current and with water dilution, the H_2 yield slightly increases, whereas the O_2 content slightly decreases, and the N_2 content changes nonmonotonically. The maximal yield of H_2 is revealed if ethanol and water in the mixture are in equal amounts.

Fig. 10 shows the dynamics of the establishment of temperature in the PLS-DGCLW reactor during the ethanol processing as measured by the thermocouple. In fact, the time of the temperature stabilization depends on the composition of work solution: In case of pure ethanol, it needs 1–1.5 min; in case of pure water, it needs 6–8 min. These data were used further in calorimetric analysis.

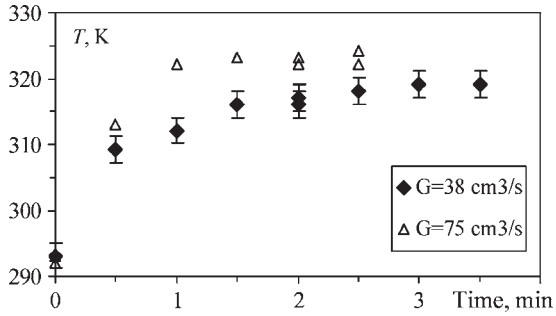

Fig. 10. Temperature of ethanol solution in the DGCLW reactor versus the processing time. $I = 200$ mA ($W = 300$ W).

III. Numerical Modeling and Results of Calculations

In numerical modeling, the composition and concentration of gas species produced in the PLS reactor with the DGCLW discharge were calculated using a system of kinetic equations for kinetically valuable components of the air–ethanol–water vapor plasma together with the Boltzmann equation for the electron energy distribution (EED) similarly to the fluid (volume averaged) model used in [27] and modified by taking into account the specific cross sections and rate constants for plasma-chemical reactions in the air–ethanol–water mixture according to recent recommendations of NIST [28]. The full kinetic mechanism includes 76 species (C_2H_5OH, H_2O, N_2, O_2, H_2, CO, CO_2, CH_4, etc.), 97 electron-impact processes, and 441 reactions (details are available at the website http://www.iop.kiev.ua/~plasmachemgroup). In calculations, it is assumed that 1) the electric power introduced into the discharge is immediately averaged in the plasma volume, 2) the electric field in the discharge does not vary in space and time, and 3) after the pass of the gas through the discharge into the liquid, its content is totally refreshed, and its flow rate in the reactor volume is the same as in the discharge gap. According to the model, reactive plasma is characterized by two temperatures, T_e and T; the electron temperature T_e is determined by the applied electric field E/N and calculated EED; the gas temperature T is determined by the temperature conditions of surrounding gas–liquid medium.

Fig. 11 shows the results of the kinetic modeling of the dynamics of formation of the basic components of synthesis gas during the processing of ethanol in the DGCLW and in the PLS reactor (calculated for $C_2H_5OH : H_2O = 1 : 1$, $I = 200$ mA, $G = 55$ cm^3/s, and $T = 323$ K). It is seen that, during the discharge time (up to $\sim 10^{-3}$ s), the concentrations of H_2, CO, and other species steeply grow up with the residence time. Then, they reach maximal values, and during the next time period from 10^{-3} to ~10 s (the time of the gas output from the reactor), they have no dramatic changes. The final transformation of CO and CO_2 at the end of the process is related to the water–gas shift (WGS) reaction

$$H_2O + CO \rightarrow CO_2 + H_2 (\Delta H = -41 \text{ kJ/mol}) \qquad (1)$$

that plays a dominant role in the H_2 production outside the electric discharge.

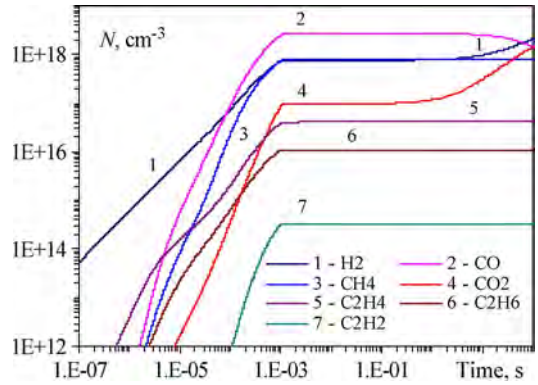

Fig. 11. Time dependences of concentrations of the synthesis gas components during the processing of ethanol in the DGCLW and in the PLS reactor at $T = 323$ K, $C_2H_5OH : H_2O = 1 : 1$, $I = 200$ mA, and $G = 55$ cm^3/s.

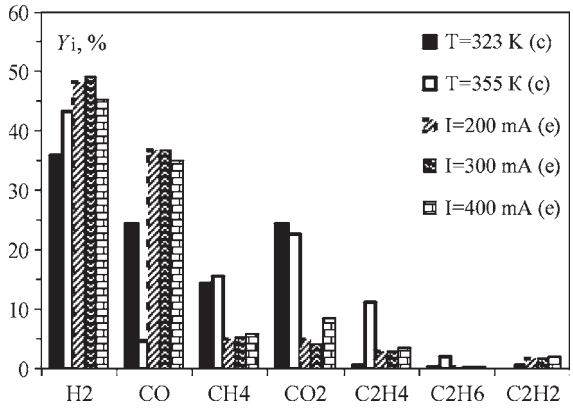

Fig. 12. Comparison of the component content of the synthesis gas products after the ethanol conversion in the PLS-DGCLW obtained at measurements and calculations.

Fig. 12 shows the summary results of the calculations of the relative concentrations of the basic components of synthesis gas at the outlet of the reactor performed for two values of medium temperature $T = 323$ and 355 K, in comparison with experimental data at different discharge currents $I = 200$, 300, and 400 mA. Despite the scatter of the data, one can see that, in the case of $T = 323$ K (as measured by the thermocouple), the calculated steady-state concentrations of the main syngas components, H_2 and CO, are close to experimental values, whereas in the case of $T = 355$ K (as assumed for the boiling in the 50% ethanol–water solution), the concentrations of syngas components differ. At that, the content of H_2 does not vary very much, because, on one side, the water vapor content in the volume increases, leading to the increase of the H_2 yield; on the other side, the time of the air flowing through the volume decreases; therefore, the H_2 yield decreases too. Under the influence of complementary factors, the H_2 content keeps practically at the same level.

IV. Analysis of Efficiency of Plasma Fuel Reforming

The estimation of efficiency of the proposed method of plasma reforming of liquid ethanol into synthesis gas in the PLS-DGCLW reactor was performed on the basis of thermochemical calculations using the following criteria: 1) energy cost of 1-m^3 syngas products; 2) productivity of conversion; 3) specific heat of 1-m^3 syngas combustion; and 4) energy efficiency. These calculations were made with taking into account standard thermochemical constants of hydrocarbons [28] using the formula for the coefficient of energy transformation

$$\alpha = \frac{\sum_i Y_i \times \mathrm{LHV}(Y_i)}{\mathrm{IPE}} \tag{2}$$

and also the parameter of efficiency from Fulcheri *et al.* [15]

$$\eta = \frac{(Y_{\mathrm{H_2}} + Y_{\mathrm{CO}}) \times \mathrm{LHV}(\mathrm{H_2})}{\mathrm{IPE} + Y_{\mathrm{HC}} \times \mathrm{LHV}(\mathrm{HC})}. \tag{3}$$

Here, IPE is the input plasma energy, Y is the molar fraction, LHV is the lower heating value of syngas components, and HC is the hydrocarbon fuel (ethanol). Equation (3) assumes that CO can be totally transformed into H_2 by the WGS process (1) with zero energy cost.

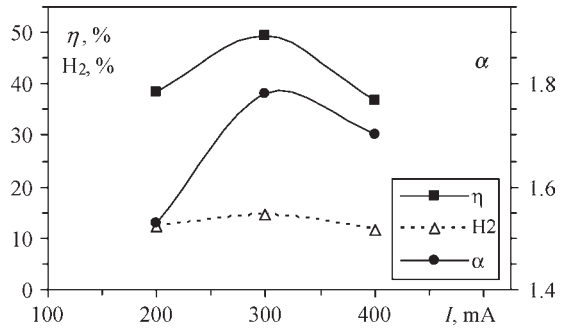

Fig. 13. Energy efficiency η, coefficient α, and H_2 yield in the process of ethanol reforming by the DGCLW.

The results of estimations in the form of $H_2(I)$, $\alpha(I)$, and $\eta(I)$ dependences are shown in Fig. 13. One can see that the net H_2 yield in the investigated discharge at $I = 300$ mA is ~15%, whereas the energy efficiency of the proposed method of ethanol conversion into syngas is up to 50%. These numbers correlate with our earlier results [25] and are comparable with other known plasma-aided ethanol reforming methods [15].

V. Conclusion

In summary, our investigations allow one to conclude the following.

1) A dynamic PLS with the electric DGCLW formed by a submerged air flow in liquid ethanol is quite efficient in the plasma-chemical reforming of ethanol into synthesis gas due to the production of chemically active agents in sufficient amount.
2) The main components of synthesis gas produced from ethanol in the DGCLW reactor are hydrogen H_2 and carbon monoxide CO, whose relative yield is many times higher than for hydrocarbons CH_4, C_2H_2, C_2H_4, and C_2H_6.
3) The composition content of synthesis gas and the power inputs on the ethanol conversion in the DGCLW depend on the gas that forms the plasma and on the ethanol–water

ratio in the solution. The H_2 yield increases with increasing discharge power and reaches the maximum if ethanol and water in the mixture are in equal amounts.

4) The minimum of the electric power input in the investigated discharge regimes is ~ 2.3 kWh/Nm^3 at the output syngas power of ~ 4.1 kWh/Nm^3.
5) The kinetic plasma-chemical modeling is in a fairly good agreement with experimental data (at least, for the main syngas components, H_2 and CO), predicting a nonthermal plasma-chemical mechanism of the ethanol conversion in the investigated PLS.

Although there is a lot of research needed before such technology can be made technically viable, this nonthermal plasma fuel reforming process looks very promising.

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Valeriy Ya. Chernyak was born in Kyiv, Ukraine, in 1948. He received the M.S., Ph.D., D.Sc., and Professor degrees from Kyiv National Taras Shevchenko University, Kyiv, in 1969, 1973, 2005, and 2008, respectively, all in physics and electronics.

He was an Assistant Professor, an Associate Professor, and a Lecturer with the Faculty of Radiophysics, Kyiv National Taras Shevchenko University, where he is currently a Professor. He is also the Head of the Plasma Chemical Research Laboratory, Department of Physical Electronics. He has authored more than 200 publications and presented many invited lectures in international conferences and workshops. His research interests are nonequilibrium plasma chemistry and its applications, particularly plasma-assisted reforming of liquid hydrocarbons.

Sergej V. Olszewski was born in Kremenchug, Ukraine, in 1965. He received the M.S. degree in physics and electronics and the Ph.D. degree in plasma physics from Kyiv National Taras Shevchenko University, Kyiv, Ukraine, in 1988 and 2000, respectively.

He is currently a Senior Research Scientist with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University. He has authored more than 30 publications. His research interests include experimental study of electric discharge in plasma–liquid systems and their applications.

Vitalij V. Yukhymenko was born in Kyiv, Ukraine, in 1980. He received the M.S. degree in physics and electronics in 2002 from Kyiv National Taras Shevchenko University, Kyiv, where he is currently working toward the Ph.D. degree with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics.

He is currently a Junior Researcher with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics. He has authored more than 30 publications. His research interest includes experimental study of plasma–chemical processes in electric discharge plasma–liquid systems and their applications.

Elena V. Solomenko was born in Kyiv, Ukraine, in 1987. She is currently working toward the M.S. degree with Kyiv National Taras Shevchenko University, Kyiv.

She is currently preparing her degree work at the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University. Her research interest includes experimental study of plasma properties of electric discharge in plasma–liquid systems.

Iryna V. Prysiazhnevych was born in Kyiv, Ukraine, in 1978. She received the M.S. degree in radiophysics and electronics in 2001 from Kyiv National Taras Shevchenko University, Kyiv, where she is currently working toward the Ph.D. degree with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics.

She is currently a Junior Researcher with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University. She has authored more than 20 publications. Her research interest includes optical diagnostics of nonequilibrium plasma of electric discharge in plasma–liquid systems.

Vadym V. Naumov was born in Loksa, Estonia, in 1954. He received the M.S. degree in energy engineering from Moscow Bauman State Technical University, Moscow, Russia, in 1978 and the Ph.D. degree in thermophysics from the Central Institute of Aviation Motors, Moscow, in 1985.

In 1986, he joined as a Member of Research Staff with the Institute of Physics, National Academy of Sciences of Ukraine, Kyiv, Ukraine. He is the Head of Photonics Research Laboratory, Institute of Fundamental Problems for High Technology, National Academy of Sciences of Ukraine (formerly Ukrainian Academy of Sciences), Kyiv. He is also an Associate Research Scientist with the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University, Kyiv. He has authored more than 200 publications. He is the holder of 18 patents. His research interests include diagnostics and modeling of nonequilibrium plasma–chemical processes in gas discharge plasma and their applications.

Dmitry S. Levko was born in Lugansk, Ukraine, in 1984. He received the M.S. degree in physics from the Vladimir Dal East-Ukrainian National University, Lugansk, in 2006. He is currently working toward the Ph.D. degree with the Institute of Physics, National Academy of Sciences of Ukraine, Kyiv, Ukraine.

He is currently a Junior Researcher with the Institute of Physics, National Academy of Sciences of Ukraine. His research interest includes numerical modeling of plasma–chemical kinetics in electric gas discharges.

Anatolij I. Shchedrin was born in Dalian, China, in 1946. He received the M.S. degree in physics from Rostov State University, Rostov, Russia, in 1969 and the Ph.D. degree in theoretical physics and the D.Sc. degree in physical electronics from the National Academy of Sciences of Ukraine, Kyiv, Ukraine, in 1972 and 1989, respectively.

He is currently the Head of the Plasma Kinetics Research Group and a Leading Scientist with the Institute of Physics, National Academy of Sciences of Ukraine. He has authored more than 100 scientific publications. His research interests include theoretical modeling of nonequlibrium kinetics of plasma–chemical processes in electric gas discharges and excimer lasers.

Andriy V. Ryabtsev was born in Kyiv, Ukraine, in 1966. He received the M.S. degree in physical engineering from Moscow Physical Technical Institute, Moscow, Russia, in 1989 and the Ph.D. degree in physical electronics from the National Academy of Sciences of Ukraine, Kyiv, in 2006.

He is currently a Research Scientist with the Department of Gaseous Electronics, Institute of Physics, National Academy of Sciences of Ukraine. He has authored more than 30 publications. His research interest is computer simulation of plasma kinetics in electric gas discharges and excimer lasers.

Valentina P. Demchina was born in Kyiv, Ukraine, 1954. She received the M.S. degree in thermochemical treatment of material from Kyiv Polytechnic Institute (Technical University), Kyiv, Ukraine.

She is currently a Senior Researcher with the Gas Institute, National Academy of Sciences of Ukraine, Kyiv. Her research interest includes gas chromatography analysis of hydrocarbons.

Vladimir S. Kudryavtsev was born in Kyiv, Ukraine, in 1959. He received the M.S. degree in aircraft construction from the Civil Aviation Institute, Kyiv, Ukraine.

He is currently a Senior Researcher with the Gas Institute, National Academy of Sciences of Ukraine, Kyiv. His research interest includes mass spectrometry analysis of gas-phase products.

Eugene V. Martysh was born in Latvia in 1952. He received the M.S. degree in physics and electronics and the Ph.D. degree in plasma physics from Kyiv National Taras Shevchenko University, Kyiv, Ukraine, in 1976 and 1983, respectively.

He is currently an Associate Professor with the Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University. He has authored more than 80 publications. He is the holder of one patent. His research interests are physics of heterophase electric discharge plasmas and their applications.

Maxim A. Verovchuck was born in Kyiv, Ukraine, in 1986. He received the M.S. degree in physics and electronics from Kyiv National Taras Shevchenko University, Kyiv, in 2008.

He is currently preparing his degree work at the Plasma Chemical Research Laboratory, Department of Physical Electronics, Faculty of Radiophysics, Kyiv National Taras Shevchenko University. His research interest includes experimental study of plasma properties of electric discharge in plasma–liquid systems.