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Autoreferát dizertačnej práce

Úprava povrchových vlastností poréznych polymérnych materiálov nízkoteplotnou plazmou pre ich použitie ako separátorov v alkalických elektrolyzéroch

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Introduction

Depletion of fossil fuel resources and environmental pollution associated with their utilisation drive the effort of governments to introduce more ***renewable energy sources*** into energy sector. However, it becomes increasingly difficult to match their ***intermittent power output*** with the demand for energy in the grid. Therefore, large-scale ***energy storage capacities*** must be available to introduce major amount of renewable power into energy mix. ***Power-to-gas*** concept represents a group of technologies dedicated to store renewable power via conversion of electrical energy into chemical energy of fuels. Such technologies are inevitable especially for those states, which cannot exploit electromechanical conversion systems, such as pumped hydro power plants. Key step in this process is the ***production of hydrogen*** by means of ***electrolysis of water***. Electrolytic hydrogen can be subsequently converted to another gaseous or even liquid hydrocarbon fuels via well establishes chemical processes. Such „green" energy carriers could play an important role in development of future energy networks, based on renewable resources. However, to promote industrial electrolyser installations and to undercut price thresholds for electrolytic hydrogen, many aspects like energy efficiency, generation capacity, capital costs and others should be taken into account. Energy efficiency and operational expenditures of electrolysis plant have direct impact on viability of electrolytic hydrogen to compete with its production from fossil fuels, which is a way that majority of hydrogen is produced today, meanwhile substitution of fossil hydrogen with renewable one represents the only roadmap for ***sustainable hydrogen economy***. Moreover, to feed the electrolysers with wind and solar power, the electrolysers must be suited for dynamic and even ***interruptible operation***.

Alkaline water electrolysis (AWE) has been developed since the beginning of 20th century and represents well established and robust electrolyser technology. Although most of existing advanced alkaline electrolysers are capable to operate in certain range of their rated power capacity, intermittent operation create higher requirements for ***inter-electrode separators*** to prevent formation of hydrogen-oxygen gas mixtures and to meet safety and ***gas purity standards***. Because mechanisms that cause mutual cross-contamination of product gases are almost independent on power loading of alkaline water electrolysis cell, decrease in gas purity is an issue especially at ***low power loading***. This is because permeation of gases through separator membrane becomes significant in respect to gas production rate.

High impurity of product gases may decrease the overall energy and cost efficiency of the process. This is because foreign gas admixture (oxygen in hydrogen and vice versa)

represent loss of electrolysis product as it is removed by recombination catalysis. Moreover, electrolysis plant has to be equipped with respective modules to perform purification.

Inter electrode separator as an integral part of AWE cell affects both energy efficiency of the electrolysis process and product gas purity. Thus, the ***development of new separator membranes*** is an active area of research.

Current status

The research in separator development has been divided into several directions, while all aim to increase ***hydrophilicity*** of the material, of which the separator is made. The reason is that porous separator must soak the liquid electrolyte to ensure its ***ionic conductivity***. In AWE, highly concentrated aqueous solution of potassium hydroxide at temperatures around 80 °C is used as electrolyte, representing highly ***caustic*** alkaline environment. First developed alkaline electrolysers incorporated ***chrysolite asbestos*** as a diaphragm due to its high wettability, porosity, well defined pore size distribution and satisfactory chemical stability in strong alkaline solution up to 80 °C. However, in 1999, asbestos has been ***withdrawn*** from commercial use in new devices.

Sintering of various inorganic powders, for example ZrO_2, was identified as an alternative approach to produce diaphragms for AWE. The main drawback of such ***inorganic diaphragms*** lies in their ***fragility*** so that high thickness is necessary, resulting into moderate or high electrical resistance.

In contrast to inorganic materials, some polymers, denoted as ***thermoplastics***, are characterised by ***mechanical strength*** and also ***high chemical resistance*** in strong alkaline solutions. Polymers including polypropylene (PP), polytetrafluoroethylene (PTFE), polyethersulphones (PES), polysulfones (PSF) and polyphenylene sulfide (PPS) are widely used for membrane fabrication. Such membranes can be used for solvents filtration and fabricated with well defined thickness, porosity, tortuosity and pore size. Porosity and tortuosity are important especially in terms of ionic conductivity, while pore diameter affect membrane transport properties. The main ***drawback*** of polymer membranes is ***hydrophobic*** surface characterized by low surface energy, which rules out their application as separators in AWE without surplus modifications.

Main stream of development has been therefore focused on development on ***polymer-inorganic composites***, which raise the advantages of both groups of materials. In these materials, polymer matrix serves as a structural support while inorganic component is used as

hydrophilicity-enhancing additive. Porous composite membranes can be prepared by a phase inversion process, where inorganic component in a form of fine powder is distributed homogenously through the whole volume of resulting composite. The inherent disadvantage of this manufacturing process lies in significant ***microstructural asymmetry*** of the membrane. Consequently, the membranes are characterised by wide pore size distribution and volumetric resistivity varies significantly through their cross-section. The majority of abundant groups of oxides such as silicates, sulphates and carbonates have higher wettability compared to polymers, but most of them are not stable in alkaline electrolyte. One of the mostly used separator in alkaline water electrolysis today is ***Zirfon® Perl***. It is a composite of PSF matrix doped with ZrO_2 filler cast into a microporous membrane. Wettability by electrolyte is achieved at high content of ***ZrO_2 filler*** (85 wt.%), which makes the membrane ***expensive*** and reduces its mechanical strength, so that it has to be reinforced by PPS mesh. Abundant minerals including wollastonite, forsterite and barite were proposed to be used as less expensive ***alternatives*** to ZrO_2, however Si-containing minerals suffer from significant ***corrosion*** in KOH electrolyte.

Anion exchange membranes (AEM) represent a different concept based on ***solid*** polymeric ***electrolyte*** with chemical and structural composition adapted to conduct OH- ions Here, electrode catalysts are deposited directly on both sides of the separator membrane, similarly to PEM electrolysis cells. The advantage of AEM electrolysis over conventional alkaline electrolysis is more ***compact cell*** design and the possibility to operate with considerably ***reduced concentration*** of KOH electrolyte. However, AEM electrolysis is still in the ***development stage*** because sufficient ionic ***conductivity*** and reasonable ***durability*** are ***hard to ensure***.

Some studies were focused on development of ***separators*** for alkaline secondary ***batteries*** by surface modification of polypropylene membranes. Namely, hydrophilic modification of microporous Celgard® was induced by ***graft polymerisation*** of ***acrylic acid (AAc)***. Such modification led to greatly enhanced electrical conductivity in 30 wt% KOH. However, despite the excellent performance of AAc grafted membranes in batteries, predominantly in terms of electrolytic resistance, ***minor effort*** has been devoted to investigate such surface modification on polymer membranes to prepare separators for AWE. Except several works dedicated to grafted PTFE membranes that were published in previous century, there are no available data on performance of membranes grafted with acrylic acid in AWE.

Objectives of the study

The concept of advanced alkaline electrolysis with zero-gap cell geometry ***sets high requirements*** on ***inter-electrode*** separators, predominantly in terms of electrical resistance gas separation efficiency and chemical durability at elevated temperatures. Permeation of H_2 and O_2 through separator becomes an issue at ***low current density*** operation, preventing AWE units to be applied in PtG systems with ***direct connection*** to renewable sources, characterised by ***intermittent power*** output.

Based on the fact that polymer membranes grafted with acrylic acid exhibited excellent conductivity in alkaline batteries along with the assumption that it could also considerably restrict diffusion of electrolysis products, the intention of this work is to investigate properties of AAc grafted PP and PES membranes in respect to operational conditions of AWE. For this purpose, objectives of the work were divided into several closely related parts:

1. Design and construction of apparatus, including gas purity analyser (TCD), for measurements of ***hydrogen/oxygen cross-contamination*** in experimental electrolysis system using various separator membranes.

2. Design and construction of apparatus for separator ***resistivity measurements*** in four-contact configuration with reference electrodes.

3. Construction and optimisation of low-pressure ***plasma reactor*** for membrane activation in radio-frequency, capacitively-coupled discharge. Construction of matched RF power supply.

4. Optimization of ***plasma-activation*** of membranes for production of hydroperoxide functional groups, which are suitable as precursors for graft polymerization.

5. Optimization of the ***graft polymerization process*** and modification of membranes by plasma-initiated graft polymerization of AAc in liquid phase.

6. ***Characterization*** of the AAc-grafted ***membranes as separators*** in alkaline electrolysis cell. Investigation of the effect of grafting degree on electrical resistance and permeability of H_2 and O_2 through the grafted membranes under electrolysis conditions.

7. Long-term ***ageing tests*** of the separator membranes in alkaline electrolyte at various temperatures.

Methods

Hydrophilic modification was performed by membrane activation in low temperature plasma with subsequent graft polymerisation of AAc in liquid phase. Polyacrylic acid is highly hygroscopic material which swells extensively in aqueous environment. It is characterised by high density of polar carboxyl groups and provides the grafted surface high wettability by polar liquid such as aqueous solution of potassium hydroxide, which is used as electrolyte in AWE.

Four types of porous substrates were examined in more detail. Three of them, made of ***polypropylene*** (PP), differed from each other in thickness, average pore size and porosity, while the fourth one was made of ***polyethersulfone*** (PES).

Prior AAc grafting, the membranes were ***activated*** in dielectric coplanar surface barrier discharge ***(DCSBD)*** or in a low pressure ***radio-frequency discharge***. The activation was optimised in terms of concentration of ***hydroperoxide*** functional groups, which were recognised as initiators of AAc graft polymerisation, as well with respect to ***mechanical strength*** of membranes. The results were used to prepare membranes with various amounts of grafted AAc (***grafting degree*** - GD) to be tested under conditions of alkaline water electrolysis cell.

The membranes were characterised in terms of ***electrolytic resistance*** in alkaline electrolyte (KOH solution) in four-contact cell configuration. The test cell comprised a pair of Hg/HgO reference electrodes, which were used to measure ohmic overvoltage across the tested separator. Area-specific resistance was determined from measured volt-ampere characteristics of the cell without separator and with inserted separator.

Separators were also characterised in terms of gas separation efficiency in AWE by measurement of ***mutual cross-contamination*** of product gases as a function of cell current density. In order to determine the diffusion flux of H_2 through separators from O_2 impurity, electrolyte was circulated by means of two identical gear pumps in two independent loops, one for cathodic and one for anodic half-cell. The measurements were conducted with the use of self-designed ***thermal conductivity detector***. The acquired data on oxygen contamination by hydrogen were used for calculations of H_2 diffusion flux density through separators and for determination of effective diffusion coefficient of H_2. Both were evaluated with respect to membrane grafting degree.

In addition, ***durability*** of surface modification was studied at ambient and elevated temperatures by gravimetric analyses, CWST tests, ATR-FTIR spectroscopy and SEM imaging.

Results

It was shown in this study that ***atmospheric plasma treatment*** of PP membranes in air was efficient in introduction of polar functional groups (predominantly carbonyl and carboxyl) onto PP surface, which led to substantial ***increase*** of their ***wettability*** by alkaline electrolyte. It resulted into reasonable ***electrolytic conductivity***, so that they could be applied as separators in AWE cell. However, surface analyses revealed degradation of the modification within several days in 30 wt% KOH solution at ambient temperature, probably caused by leaching of the whole oxidised layer from the polymer surface into alkaline solution.

In order to achieve better stability of surface modification, plasma activation followed by graft polymerisation of acrylic acid was studied in more detail. Plasma ***activation*** in low-pressure, capacitively-coupled discharge at frequency of 13.56 MHz was ***optimized*** in terms of working gas, power and exposure time. The experiments on membrane activation were conducted with the use of two different power supplies, while the second (enhanced) version is presented in Fig. 1. It can be seen that RF power supply consisted of signal generator and MOSFET-based amplifiers. The supply was connected to the vacuum plasma reactor through impedance matching network. In addition, the RF power supply was designed to offer pulsed operation by means of timer attached to enable inputs of amplifiers. Pulsation was introduced in order to modulate discharge power by means of variation of generator duty cycle.

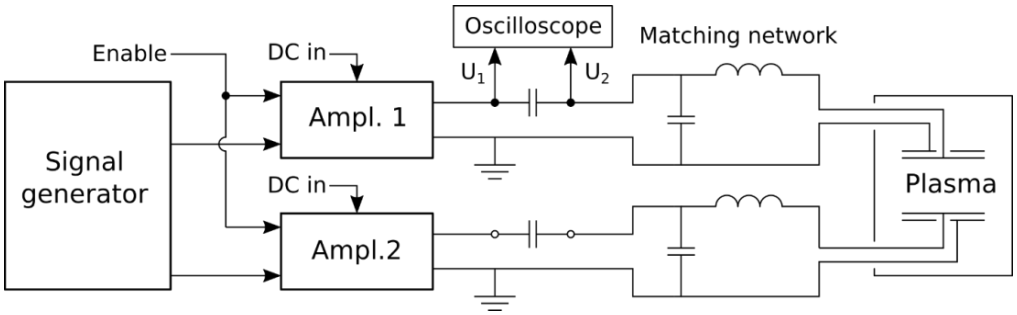

Fig. 1: Schematic diagram of low pressure plasma reactor

The dependence of hydroperoxide concentration and ultimate tensile strength of membranes activated in low-pressure O_2 plasma at power of 12.5 W is presented in Fig. 1(left), whereas evolution of the same parameters in respect to generator duty cycle (plasma power) at 180 s exposure is presented in the Fig. 2 (right).

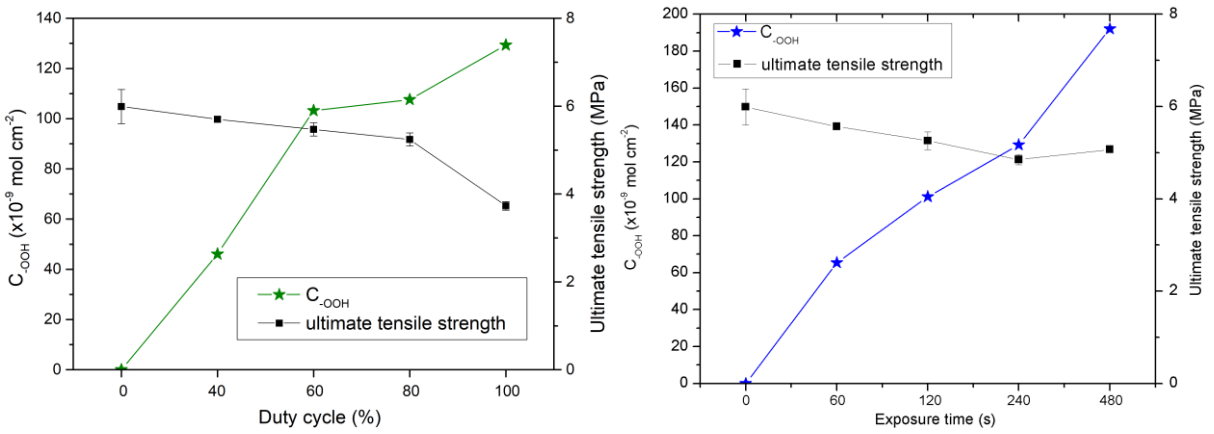

Fig. 2: Optimisation of plasma activation in low-pressure discharge in oxygen

The study brought the following observations:

- ***Oxygen*** plasma was ***more efficient*** in production of hydroperoxide functional groups than Ar plasma under studied plasma parameters.
- In pulsed regime at 12 kHz and generator duty cycle of 50 %, ***hydroperoxide*** concentration increased ***monotonously*** with ***exposure*** up to 480 s.
- ***Plasma exposure*** had ***no*** significant ***effect*** on ***mechanical strength*** of PP membranes at 50 % duty cycle, but mechanical strength decreases slightly with increasing generator duty cycle at 180 s exposure.

The subsequent AAc grafting, conducted in liquid phase, led to formation of AAc graft co-polymer through the whole cross section of membranes and to various grafting degrees, depending on the grafting parameters and substrate. In case of PP membranes, grafting was associated with membrane thickening due to swelling of pAAc. Contrary, ***no change in dimensions*** or shape of PES-A membranes was observed. As an example, SEM images of PP-A and PP-Celgard 2400 at magnification of 3 kx and 30 kx, respectively are shown in Fig. 3

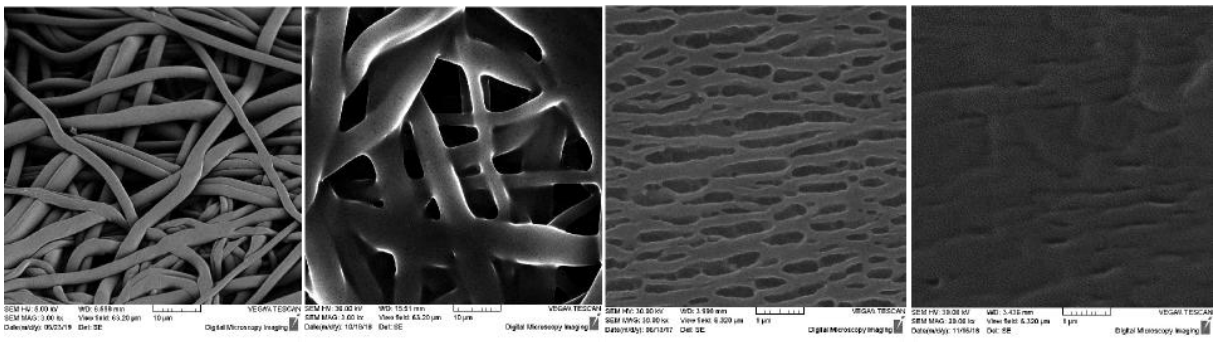

Fig. 3: SEM images performed on Tescan Vega II of a) untreated PP-A, b) AAc-grafted PP-A, c) untreated Celgard 2400 and d) AAc-grafted Celgard 2400

The grafted pAAc was neutralized in KOH solution and formed a potassium carboxylate salt. The membrane wettability by KOH electrolyte was reflected by enhanced electrolytic resistance and it was concluded that:

- In case of Celgard 2400 and PES-A the increase in GD led to substantial reduction of electrolytic resistance.
- Increasing GD on PP substrates with fibrous structure (PP-A and PP-B) was associated with minor changes in resistance at higher GD due to membrane thickening, yet volumetric resistivity became decreased.
- Untreated PP membranes could not be characterised in AWE cell because of negligible conductivity.

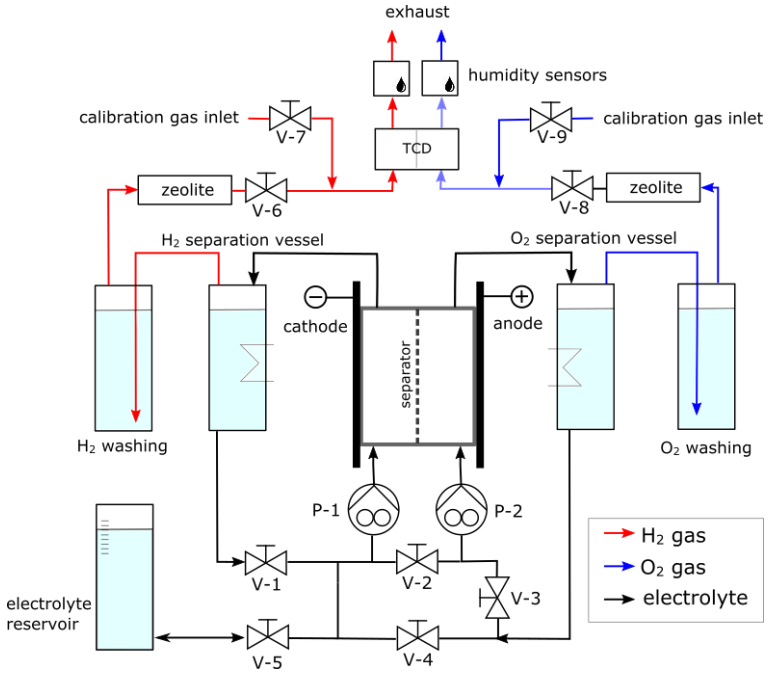

Fig. 4: Electrolysis system for characterisation of membranes in terms of gas separation efficiency

The membranes were further investigated in terms of gas separation efficiency in AWE cell. They were tested in electrolysis system with separated electrolyte loops, shown in fig. 4, where diffusion through separator is the only mechanism of gas cross-contamination. With all studied types of substrates except of Celgard 2400 it was shown, that ***increasing GD reduces H_2 diffusion flux density*** j_{H2} through membranes. The increase of j_{H2} through Celgard 2400 was concluded to be caused by membrane wrinkling at higher GD, thus increased membrane effective area. The below-included Tab. 1 summarizes the best achieved performance of prepared membranes in terms of electrolytic resistance and anodic hydrogen concentration at cell current density of 50 mA cm^{-2} using 30 wt% KOH electrolyte at 50 °C.

Tab. 1: characteristics of modified membranes in AWE

separator	pore size (µm)	porosity (%)	thickness (µm)	GD (wt. %)	resistance ($m\Omega cm^2$)	anodic H_2 (vol.%)
PP - A	1 um	40	315	40	201	0,070
PP - B	0.45 um	33	322	70	273	0.042
Celgard 2400	-	39	25	177	42	0.215
PES - A	1 um	72	140	21	20	0.045

In Fig. 5 it can be seen, that the ***ratio*** of H_2 ***diffusion flux*** density to membrane ***specific conductivity*** was observed to ***decrease monotonously*** with GD for all membranes under tests, which justifies that overall performance of membrane improves with increasing GD. It should be noted however, that supersaturation of electrolyte by dissolved species would conceivably lead to ***higher*** diffusion fluxes in case the separators were used in ***zero-gap cell geometry***.

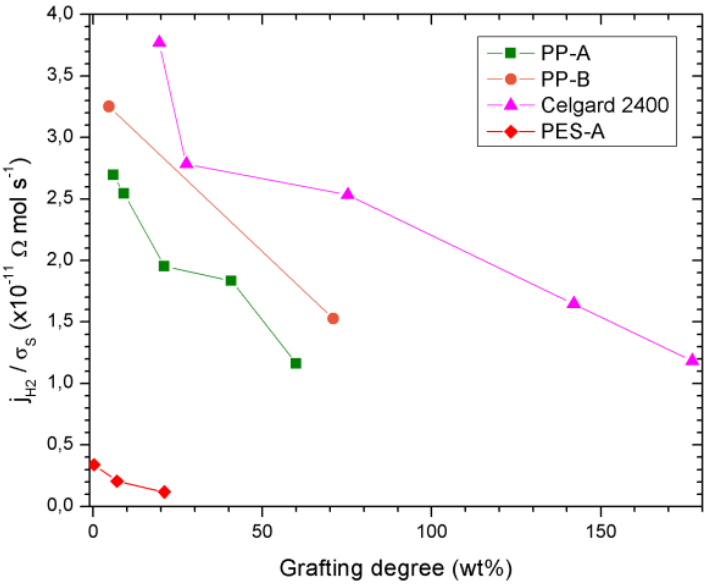

Fig. 5: Ratio of H2 diffusion flux through membrane to membrane specific conductivity as a function of AAc grafting degree.

Ageing tests revealed that storage of grafted membranes in 30 wt% KOH

- was accompanied with gradual weight loss at 60 °C, but retained hydrophilicity for at least 8500 hours.
- led to degradation of PP substrate grafted with AAc at 80 °C within less than 800 h

Deterioration of polymer backbone and release of PP chains together with grafted AAc co-polymer into KOH solution was suspected to be one of the possible explanation of such

ageing effect. However, preliminary results show that PP substrate without treatment could survive 80 °C KOH without major decrease of mechanical strength for more than 2000 h. It is plausible to assume, that further optimisation of plasma activation would reduce substrate degradation during long term exposure to hot KOH electrolyte.

Summary

The results presented in the work reveal that AAc graft co-polymer greatly enhances electrical conductivity of PP and PES porous membranes and limits considerably the gas crossover that is caused by diffusion of electrolysis products dissolved in the KOH electrolyte. In particular it was shown, that the ***ratio*** of ***H_2 diffusion flux*** to membrane area-specific ***conductivity decreases monotonously*** with amount of AAc graft copolymer for PP and PES membranes studied in this work. The lowest electrolytic resistance achieved within this study was ascribed to PES membrane with GD of 21 wt%. It accounted for 20 mΩ cm^{-2} in 30 wt% KOH at 50 °C, which is much lower than values reported for state of the art Zirfon® Perl composite separator. Moreover, it was shown that in the present cell geometry H_2 diffusion flux determined for grafted PP or PES membranes is comparable or even lower than values obtained in other works with Zirfon separator. However no direct comparison could be made because differential concentration of dissolved gases between half cells can vary significantly with cell design. Therefore, evaluation of diffusion crossover through grafted membranes in zero-gap cell geometry and its comparison to commercially available separators requires further investigation.

The reduction of diffusion of electrolysis products through separator is important especially at low power loading of electrolysis cell, where it can lead to safety hazards due to creation of gas mixtures close to flammability limits. The results presented in this work are considered to be valuable for ***development of alkaline electrolysers***, especially those intended to be supplied by ***renewable power sources***, which are generally characterised by intermittent power output.

List of publications

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Registrované v: wos
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IF (JCR) 2018=4.084

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AGJ Autorské osvedčenia, patenty, objavy

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BEE Odborné práce v zahraničných zborníkoch (konferenčných aj nekonferenčných)

BEE01 Medvecká, Veronika [UKOMFKEF] (25%) - Mošovská, Silvia (25%) - Mikulajová, Anna (15%) - Omasta, Samuel [UKOMFKEF] (15%) - Staňo, Ľubomír [UKOMFKEF] (15%) - Zahoranová, Anna [UKOMFKEF] (5%): Low-temperature atmospheric pressure plasma in sproutsdecontamination
Lit.: 6 zázn.
In: Frontiers in Redox Biology and Medicine 8th FiRBaM. - Greifswald : Leibniz-Institut für Plasmaforschung und Technologie, 2019. - S. 20-20
[Young Professionals Workshop on Plasma-Medicine 2019. 8, Greifswald, 05.08.2019 - 08.08.2019]

BFB Abstrakty odborných prác z domácich podujatí (konferencie, ...)

BFB01 Stano, Michal [UKOMFKEF] (50%) - Staňo, Ľubomír [UKOMFKEF] (50%): Formation of hydroperoxide functional groups on polypropylene surface by low temperature plasma treatment
Lit.: 2 zázn.
In: Book of Abstracts: The Seminar on Trends in Plasma Physics. - Bratislava : Katedra experimentálnej fyziky, 2018. - S. 19-19
[New Trends in Plasma Physics 2018 : Seminár BB. Doľany, 04.10.2018]

Štatistika kategórií (Záznamov spolu: 16):
ADC Vedecké práce v zahraničných karentovaných časopisoch (1)
AED Vedecké práce v domácich recenzovaných vedeckých zborníkoch, monografiách (1)
AFC Publikované príspevky na zahraničných vedeckých konferenciách (1)
AFD Publikované príspevky na domácich vedeckých konferenciách (1)
AFG Abstrakty príspevkov zo zahraničných vedeckých konferencií (7)
AGJ Autorské osvedčenia, patenty, objavy (3)
BEE Odborné práce v zahraničných zborníkoch (konferenčných aj nekonferenčných) (1)
BFB Abstrakty odborných prác z domácich podujatí (konferencie, ...) (1)

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