Hydrogel alkaline electrolytes for Ni-MH batteries

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Introduction

Several years ago chemical power sources supplied power exclusively with application of liquid electrolytes that have high ionic conductivity, thus allowing quick ion migration between opposite side electrodes, which enables obtaining large amount of electricity that can be given or stored in chemical power source per unit time [1].

Apart from advantages, liquid electrolytes have also numerous disadvantages. Using liquid electrolytes in chemical power sources can be dangerous both for its direct user, as well as for the environment. Hazard of electrolyte spill in form of concentrated inorganic compounds (acids, bases), sometimes even spill of organic solvents that can be used as constituents, low electrochemical stability, inability to miniaturize the battery, or inability to change manner of its use, are serious disadvantages of liquid electrolyte applications in primary and secondary batteries [1 – 3]. They all partially result from the need to keep the cell in the vertical position.

In 1978, the team of Prof. Armand has proven that liquid electrolyte in batteries can be successfully replaced with polymer electrolyte [4], which has at the same time high ionic conductivity, thermal and mechanical stability and high operational safety. Moreover, application of polymer electrolyte allows complete elimination of separator from the electrochemical system, as it forms itself specific interelectrode barrier that prevents direct electron flow between opposite sign electrodes, which is not prevented by liquid electrolyte and moreover, it enables migration of ions between electrolytes [5 – 7].

Hydrogels can have various physical forms, starting from solid powders, microparticles, films or membranes up to solid or liquid capsules. They are made of polymer chains interconnected in various manner thus forming 3D cross-linked structures known as polymer networks [8 – 11]. However, the main component of hydrogel is water constituting 40% to 99% of its total mass. They can be produced using many different methods, while chemically cross-linked hydrogels exhibit higher thermal and chemical resistance in comparison with hydrogels obtained by means of physical cross-linking [8].

Within the structure of cross-linked hydrogel various interactions between individual system components can be distinguished. The following types of interactions can be observed: single covalent bonds, multiple bonds and connections resulting from entanglement of polymer chain [9, 10]. In dry conditions, polymer chains are irregular (Fig. 1b). As a rule, polymer chains form so-called ball, closely packed due to the high degree of entanglement of constituent individual polymer chains. In the presence of solvent, i.e. water for hydrogels, functional groups in chains are subject to solvation and dissociation, which leads to loosening of the bundle thus allowing water absorption by such a system up to a moment of maximum extension of polymer chains that form the 3D network of hydrogel (Fig. 1a) [11].

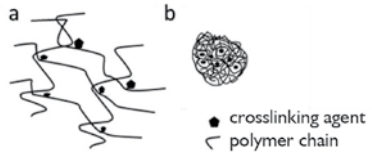

Fig. 1. Polymer chain connection diagram: a) 3D network, b) polymer ball

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In the hydrogel structure obtained through physical cross-linking by cyclic freezing and unfreezing of the PVA+H_2O mixture, water molecules are present in two forms, i.e. as bound water that is not frozen out and water that can be frozen out, i.e. free water. Water that does not crystallize at temperatures below 0°C does not undergo freezing out, while the crystallizing water does. Such water behaviour in the hydrogel structure is determined by interactions between water molecules and polar hydrophilic groups of polymers and capillary condensation phenomenon as well as formation of clusters in polymer bonds [11].

Hydrogels that can be used as polymer electrolytes in batteries are 3D cross-linked polymer matrices containing compounds of ionic structure embedded during the cross-linking process and able to transport ions between electrodes in the electrolytic system [12 – 14]. The application of such alkaline hydrogels, particularly if produced in form of polymer membranes, enables an improvement battery operation safety due to the fact that spill of corrosive (alkaline, or acidic) electrolyte is impossible and makes battery manufacturing process simpler due to the elimination of one of design components, i.e. separator.

As mentioned above, electrolyte, next to cathodes and anodes, is one of the main design components of state-of-the-art devices for energy storage and conversion and is an important research field, for material engineering, polymer chemistry and electrochemistry [5–7]. This has prompted publication of research results on possibilities of application of physical cross-linking method [15, 16] on mixture containing various weight ratios of polymer (PVA), water and KOH for production of alkaline hydrogel electrolytes for application in Ni-MH cells.

Experimental part

2 g of poly(vinyl alcohol) (PVA, VWR, M_w 1150.00) was dissolved in 25 cm^3 of water in 100 cm^3 glass container immersed in 80°C water bath, where container content was stirred vigorously for approx. 4 hours. Potassium hydroxide (KOH, POCH) was added in the following weight ratios to the PVA aqueous solution prepared in such a manner: 0:100, 20:80, 30:70, 40:60 and 50:50, in relation to PVA. Then, air bubbles were removed using vacuum from the highly viscous homogeneous solution. The solution prepared in such a manner, free of gas phase bubbles, was poured onto flat surface of plastic mould and subjected to physical cross-linking involving freezing and unfreezing of the system with four cycles in temperature range of -20 to +20°C and temperature change rate of 12°C/min. Thickness of formed in such a manner alkaline hydrogel electrolytes in form of membranes was in a range of: 0.05-0.1 cm. For the obtained polymeric alkaline hydrogel electrolytes were analysed structurally by means of molecular absorption spectroscopy. Measurements were performed using Bruker Equinox 55 IR spectrometer. Furthermore, electrolytes were subjected to the analysis of topographic structure using Scanning Electro Microscopy (SEM) that allows identification of morphological structure and determination of mean size of pores formed during the production of polymer membranes. Measurements were carried out using ZEISS EVO®40 measurement apparatus.

Measurements of conductivity of alkaline hydrogel electrolytes were conducted using Electrochemical Impedance Spectroscopy (EIS). The measurement system comprised two-electrode Swagelok-type

cell, where between two blocking electrodes of 1.27 cm^2 surface area, disc of alkaline hydrogel polymer electrolyte of the same diameter and known thickness was placed.

Values of specific conductivity of studied hydrogel electrolytes were determined from Nyquist plot and mathematical dependence between resistance and dimensions of the tested electrolyte: $\sigma = l/R*A$,

where l – thickness of hydrogel membrane, cm; A – membrane surface area, cm^2; R – electrolyte resistance determined from Nyquist plot. The measurement was conducted using the equipment VMP3, BioLogic. Impedance spectrum was measured at 25°C, for a frequency range of 0.1 Hz to 100 kHz for potential amplitude equal to 0.01 V.

Water content in alkaline hydrogel membrane was determined by weighing the membrane during its drying process. In studies, hydrogel electrolyte fragments of known weight were used and then dried at 40°C till reaching constant weight of the membrane. Water content was assumed to be equal to weight loss calculated using the following formula:

$$\% \text{ water} = \frac{m_n - m_s}{m_n} 100\%$$

where: m_n – weight of alkaline hydrogel membrane before drying; m_s - weight of alkaline hydrogel membrane after drying.

For selected alkaline hydrogel electrolytes, performance characteristics in flexible alkaline cells of pouch-type was measured. Tests were conducted in two-electrode systems of type:
(-) 90% AB_5, 10%Ni alkaline hydrogel electrolyte $Ni(OH)_2$/NiOOH (+)

Positive electrodes were made using battery mass of tradename NICOL G extra heavy (Bochemie). Active mass of negative electrode was a mixture of powder-alloy reversibly absorbing hydrogen of AB_5 type (Auersrore IV: $MmNi_{3.55}Al_{0.3}Mn_{0.4}Co_{0.75}$) with 10% nickel carbonyl addition. Weighed mixtures of masses, positive and negative, were formed into a pellet using hydraulic press with pressure force of approx. 2 kN obtaining pressure of approx. 5 MPa, which was then placed and enclosed in nickel mesh serving as a current collectors of the electrode. After electrodes had been produced, their surface area was 3.8 cm^2. Positive and negative electrodes prepared in such a manner were then subjected to preactivation in order to increase catalytic properties of alloy electrode material in reaction of hydrogen electrosorption that occurs in the cell. The process of chemical activation involved etching of electrode surface in order to remove contamination from their surface. The electrodes were immersed in 6M KOH of density equal to $d = 1.25 \pm 0.01$ g/cm^3. Similar operations were conducted for the separator of the cell with 6M KOH that was treated as a reference sample for the tested systems. The separator was an absorbent nonwoven Viledon FS2119 (Freudenberg). Electrode and separator activation were conducted for 24 h (T= 25°C, p= normal pressure, approx. 1014hPa) directly before assembling the batteries, where after activation, the excess electrolyte was removed gently from surfaces of electrode and separator using tissue paper. Then, system of two electrodes with placed between them alkaline hydrogel electrolyte of known KOH content was placed inside the pouch made of polypropylene film. The cell was closed using vacuum sealer forming in this manner flexible pouch-type Ni-MH cell.

Electrical tests of model batteries using alkaline hydrogel electrolytes involving galvanostatic, cyclic charging/discharging of tested electrochemical system were carried for current of 30 mA/g expressed as per active mass of negative electrode. The battery was charged to voltage of 1.55 V. Then, after one hour relaxation period, the tested battery was discharged to potential of 1.00 V.

Results and Discussion

During the studies, polymeric hydrogel electrolytes of the following composition were prepared (Tab. 1):

Table 1

Composition of alkaline hydrogel electrolytes

Acronyms of alkaline hydrogel electrolytes	PVA % w/w	KOH % w/w. vs PVA
PVA/0%KOH	100	0
PVA/20%KOH	80	20
PVA/30%KOH	70	30
PVA/40%KOH	60	40
PVA/50%KOH	50	50

All alkaline hydrogel electrolytes (Tab. 1) were prepared by means of physical cross-linking by cyclic freezing and unfreezing of mixtures of appropriate composition according to the procedure described in the experimental part. Hydrogel electrolyte without KOH (PVA/0%KOH) had form of a clear transparent membrane. Other alkaline hydrogel electrolytes had form of white translucent membranes of high degree of plasticity comparable for all hydrogels of 20-50% w/w KOH content in relation to PVA, in individual systems.

SEM images of surfaces of alkaline hydrogel electrolytes taken using scanning electron microscope are presented in Figure 2. Membrane surfaces that are alkaline hydrogel electrolytes have high porosity. Membrane fractures, obtained after freezing in liquid nitrogen show their internal structure. There are visible skin layers and porous internal layer containing numerous pores of various shapes and dimensions. Section images of membranes produced using KOH show also potassium hydroxide aggregates, which can be found both on surface, as well as inside pores of individual membranes. This indicates, among others, high dispersion of KOH within the structure of hydrogel electrolytes.

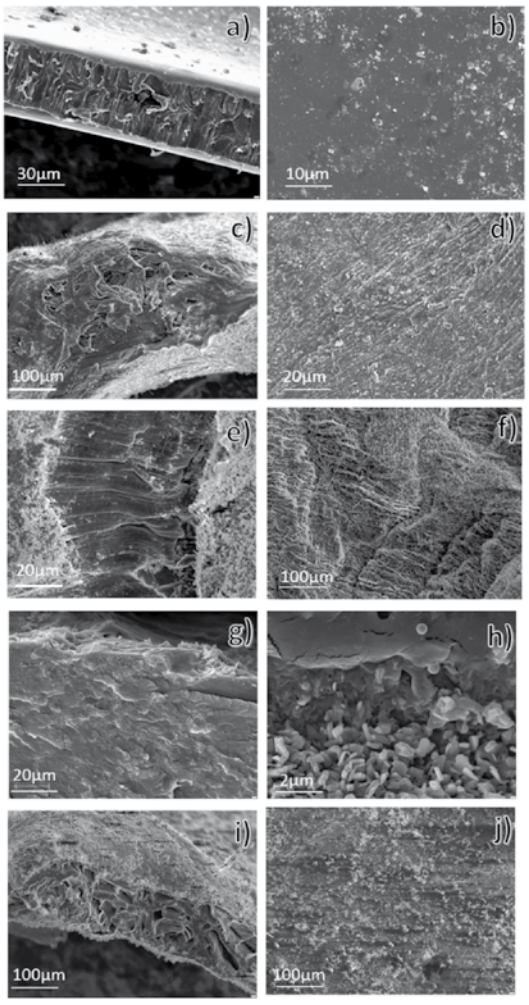

Fig. 2. SEM images of hydrogel electrolyte surfaces a), b) PVA /0%KOH, c), d) PVA/20%KOH, e), f) PVA/30%KOH, g), h) PVA /40%KOH, i), j) PVA/50%KOH

FT-IR spectra for individual polymer hydrogel electrolytes (Fig. 3) confirm composition of individual hydrogel electrolytes. Visible peak shifts indicate change in 3D structure of cross-linked polymer, depending on the KOH content in the membrane.

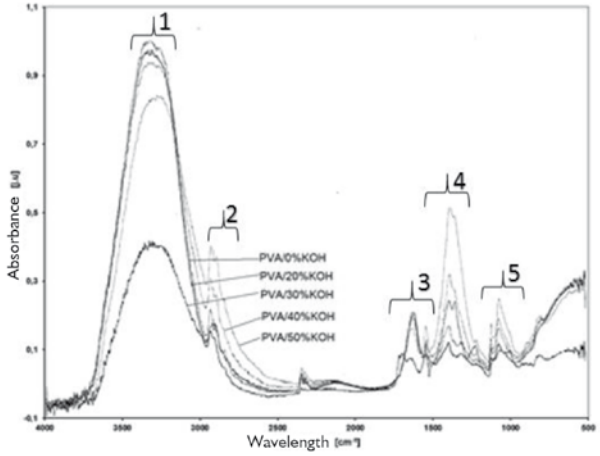

Fig. 3. FT-IR spectra of alkaline hydrogel electrolytes (1) stretching vibrations of -OH groups; 2) C-H stretching, 3) C-H scissoring, 4) C-H rocking 5) CH_2–OH stretching)

In all hydrogel electrolytes produced using PVA by means of physical cross-linking (Tab. 2) over 70% water content was found. The highest water content is in hydrogels without addition of potassium hydroxide (above 90%). Other hydrogel electrolytes (with KOH) have 75-80% w/w water content. Differences between water content in hydrogels are caused probably by the presence of KOH, which after removal of solvent, stays in the membrane.

Table 2

Water content in hydrogel electrolytes

Alkaline hydrogel electrolyte	H_2O content, % w/w
PVA/0%KOH	>90%
PVA/20%KOH	80.45
PVA/30%KOH	76.48
PVA/40%KOH	76.80
PVA/50%KOH	74.65

Based on the analysis of measurement results obtained by means of impedance spectroscopy (Fig. 4) for electrochemical systems of type: steel electrode | alkaline hydrogel electrolyte | steel electrode one can obtain information regarding properties of tested electrolytes, including electrolyte resistance – R_b. After taking into account thickness of individual electrolytes and corresponding values of R_b, specific conductance values of alkaline hydrogel electrolytes using the formula presented in the experimental part (Tab. 3).

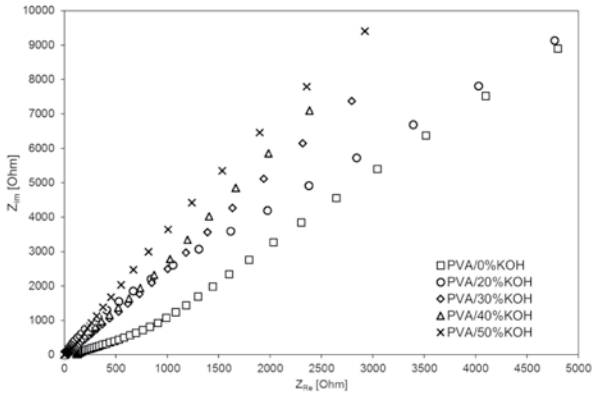

Fig. 4. Nyquist plots for hydrogel membranes of various KOH content, temp. 25°C

Table 3

Specific conductance values for investigated alkaline hydrogel electrolytes at 25°C

Alkaline hydrogel electrolyte	σ_{25}, S/cm
PVA/0%KOH	5.22×10^{-5}
PVA/20%KOH	2.68×10^{-2}
PVA/30%KOH	5.90×10^{-2}
PVA/40%KOH	13.20×10^{-2}
PVA/50%KOH	13.20×10^{-2}

It was found that the highest values of specific conductance at 25°C are exhibited by electrolytes containing 40% w/w of KOH. Increase of KOH percentage in the studied electrolytes to 50% has no effect on increase of specific conductance of hydrogel.

In comparison to typical electrolyte for Ni-MH, i.e. 6M aqueous KOH solution of specific conductance @25°C equal to 59.5×10^{-2} S/cm, specific conductance values for alkaline hydrogel electrolytes PVA/KOH are approx. 4.5 times lower. However, in terms of chemical power source handling and operation safety, alkaline hydrogel electrolytes have a significant advantage over liquid electrolyte, despite lower ion migration capability in an electrolytic system.

Based on the conductance measurements, two alkaline hydrogel electrolytes of highest specific conductance were selected providing the best possibilities of ion transport in an electrolytic system. These studies allowed very general, but useful at this stage of research, verification of potential possibility of practical application of this type of hydrogel electrolytes in the future. Value of specific conductance directly proves the possibility of ion transport in an electrolytic system with a given electrolyte, therefore it was concluded that hydrogel electrolytes of lower σ will not satisfy the expectations and are not included in the planned studies of electrical cells.

Nickel-hydride cells were assembled (Fig. 5) using alkaline hydrogel electrolytes of best performance in terms of electric conductance, i.e. systems containing 40% and 50% w/w of KOH in relation to PVA and electrodes prepared in advance.

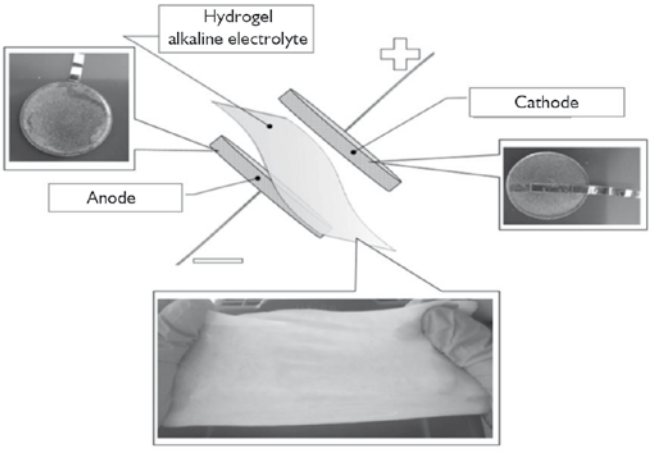

Fig. 5. Assembly of flexible pouch-type Ni-MH cells using polymeric hydrogel electrolytes

Among cells with hydrogel electrolytes, the highest discharge capacity (174 maH/g) was exhibited by pouch-type Ni-MH cells with PVA/40%KOH hydrogel alkaline electrolyte showing the highest specific conductivity among the tested hydrogel electrolytes. Moreover, it was found that tested cells containing liquid electrolyte, 6M KOH, have almost 20% lower discharge capacity equal to 143 mAh/g. Furthermore,

they exhibit higher charging voltage (Fig. 6) and lower discharging voltage (Fig. 7) than pouch-type cells with PVA/40%KOH hydrogel electrolyte. Deterioration of performance of cell with liquid 6M KOH is very likely caused by the design of the studied cell, and to be more exact, by the resulting from the design impossibility to supply sufficient amount of electrolyte in form of liquid phase. As presented in the experimental part of this paper, the cells were tested in form of flexible pouch-type systems. The design of such cells is significantly different from traditional, well-known rigid prismatic and cylindrical cells. In the flexible pocket pouch-type cell, electrodes are placed alternately separated with separator/electrolyte forming electrode stack which is subsequently closed with vacuum sealer with simultaneous removal of potential liquid phase from the inside of the cell. For typical Ni-MH cell, both electrodes and separator for the entire lifetime of the cell are completely immersed in aqueous electrolyte solution and the cell housing is closed tightly – hot sealed. The studies have shown that use of seperator typical for commercial Ni-Mh and liquid electrolyte in flexible pouch-type cells is technologically undesirable and does not allow for correct electrochemical in the cell to occur. As it is impossible to use excess electrolyte in this type of design, it cannot work properly and all attempts to introduce excess liquid phase to pouch-type cell do not allow for production of flexible and elastic Ni-MH batteries.

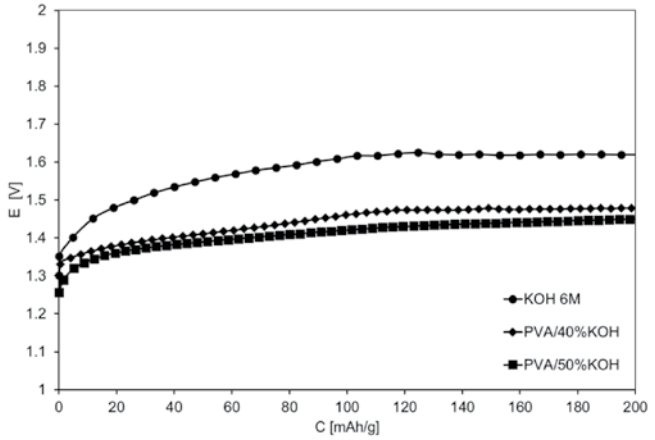

Fig. 6. Charging curves recorded for pouch-type Ni-MH cells with liquid electrolyte and hydrogel electrolytes of various composition

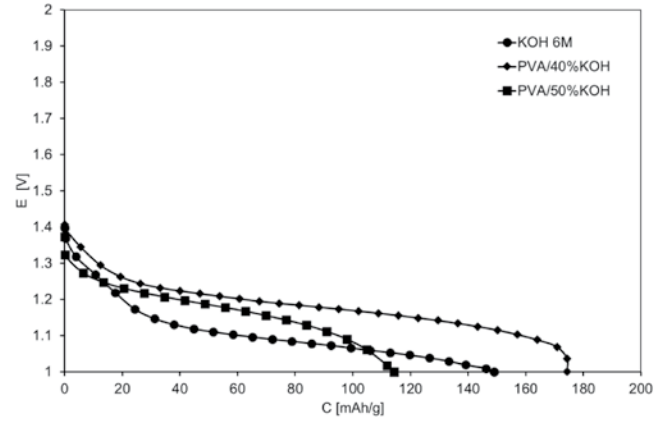

Fig. 7. Discharging curves recorded for pouch-type Ni-MH cells with liquid electrolyte and hydrogel electrolytes of various composition

During the cyclic charging and discharging, due to general research nature of our studies, cells with alkaline hydrogel electrolytes were subjected only to five full charge/discharge cycles (Fig. 8). It was observed that the highest stability of cell capacity is shown by the systems containing hydrogel electrolyte PVA/40%KOH. They have higher capacity and better operation stability during cyclic charging and discharging of the cell, even in comparison to cells with liquid electrolyte, 6M KOH, which is known to have higher specific conductivity.

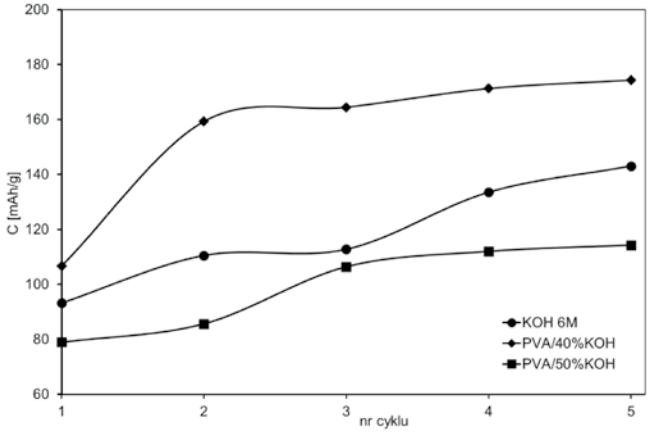

Fig. 8. Effect of number of charge/discharge cycles of battery on capacity of Ni-MH cells

Summary and Conclusions

In summary, it can be said that:

- Alkaline hydrogel electrolytes produced by means of physical cross-linking using poly(vinyl alcohol) and potassium hydroxide have a dual role in tested Ni-MH cells, i.e. that of electrolyte and of barrier.
- Alkaline hydrogel electrolytes containing 40% w/w KOH and 60% w/w PVA exhibit better electrochemical and mechanical properties in model pouch-type Ni-MH cells, which results in high discharge capacity equal to 174 mAh/g. This capacity is approx. 20% higher than capacity of similar cell with liquid electrolyte 6M KOH. Similarly, charging voltage is lower and discharging voltage is higher than for the cell with liquid electrolyte.
- Cells with alkaline hydrogel electrolytes containing 40% w/w KOH and 60% PVA show good operation stability, determined based on cyclic charge/discharge processes. This stability is higher than for cells filled with liquid electrolyte, 6M KOH.

Common application of hydrogel electrolytes would improve safety of handling of this type of cells and allow for application other than vertical electrode systems as it has place for cells with liquid electrolyte.

Source of founding

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POPULARYZACJA NAUKI

W Kielcach otwarto Energetyczne Centrum Nauki

Jak działa elektrownia? W jaki sposób można pozyskać energię z naturalnych źródeł? Jaka siła tkwi w wodzie? – m.in. na te pytania odpowiedzi znajdą odwiedzający nowo otwarte Energetyczne Centrum Nauki. Wszystko dzięki interaktywnym modelom i laboratorium. Centrum dedykowane jest nie tylko dla najmłodszych „naukowców", ale także dla ich rodziców i wszystkich ciekawych świata – przekonywali pomysłodawcy i twórcy Energetycznego Centrum Nauki otwartego w Kieleckim Parku Technologicznym. *(kk)*

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KONKURSY, NAGRODY, WYRÓŻNIENIA

Grad medali dla polskich wynalazców na targach BRUSSELS INNOVA

15 medali złotych ze specjalnym wyróżnieniem jury, 30 medali złotych, 19 medali srebrnych i jeden medal brązowy oraz liczne nagrody i wyróżnienia to dorobek polskich naukowców i wynalazców, przywieziony z 64. Światowych Targów BRUSSELS INNOVA 2015. W tegorocznej edycji Targów, poświęconych transferowi technologii i wdrażaniu postępu technicznego, zgromadzono rozwiązania z: Belgii, Chorwacji, Francji, Iranu, Malezji, Malty, Maroka, Mołdawii, Polski, Rosji, Rumunii, Tajlandii, Tajwanu i Włoch. Polscy wystawcy zaprezentowali 71 wynalazków. *(kk)*

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MAB: szansa na dofinansowanie badań naukowych i międzynarodowej współpracy

Międzynarodowe Agendy Badawcze (MAB) to projekt, który umożliwi stworzenie w naszym kraju światowej klasy ośrodków naukowych. W ogłoszonym właśnie konkursie, jednostki realizujące MAB będą mogły otrzymać łączne dofinansowanie w wysokości 530 mln PLN. MAB wzorowany jest na programie Komisji Europejskiej Teaming For Excellence organizowanym w ramach Horyzontu 2020. Najlepsze ośrodki z Unii Europejskiej mogły ubiegać się o dofinansowanie na stworzenie wiodących centrów badawczych i rozwinięcie strategicznej współpracy z zagranicznymi partnerami. Wśród laureatów pierwszego etapu, którzy otrzymali 500 tys. EUR, znalazły się trzy polskie jednostki. W ramach MBA o dofinansowanie polskich ośrodków zaangażowanych będą mogli ubiegać się wybitni naukowcy z Polski i zagranicy. Ich zadaniem będzie zorganizowanie i prowadzenie badań wspólnie z wiodącym ośrodkami z innych państw. W zwycięskich jednostkach wprowadzone zostaną najlepsze światowe praktyki z zakresu m.in. polityki personalnej, zarządzania pracami B+R oraz komercjalizacji wyników badań. *(kk)*

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