

# Ion-selective electrodes – classical systems and new ideas

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## Introduction

Ion-selective electrodes (ISEs) belong to the oldest group of chemical sensors, i.e. potentiometric sensors, where the recorded voltage value (electrode potential) is a function of concentration of a chemical compound in the sample solution. Ion-selective electrodes, as suggested by their name, enable concentration measurements (more exactly – measurements of activity) of selected ions (called primary ions) in solution, in the presence of other ions (called interferent ions) [1, 2]. The well-known and most often used kind of ISE is glass electrode, used to measure pH value in aqueous solution, in a wide range from 0 to 10 – 12, depending on the membrane composition. The idea of this electrode was proposed over 100 years ago, in 1909, by Fritz Haber and Polish scientist Zygmunt Klemensiewicz.

Nowadays ion-selective electrodes are a routine tool of chemical analysis, applied in monitoring systems used in industry, in clinical analysis to determine selected ions in blood, in environmental analysis. A similar operation principle is used in oxygen sensors mounted in cars – this device measures the voltage, and thus determines the oxygen amount in combustion gases, then basing on these results it controls the composition of the fuel-air mixture supplied to the car engine.

The principle of measurements using ion-selective electrodes is very simple, it is based on measurements of electromotive force of a cell composed of two electrodes, immersed in the sample solution. One of this electrodes is called indicator electrode, its potential is selectively dependent on concentration of given ions. The second electrode is the reference electrode, characterized by stable potential, independent of composition and concentration of the sample. A significant requirement concerning the voltage meter is its high input impedance, much higher than for typical voltmeters. This is important due to usually high resistance of ion-selective membranes, e.g. glass electrodes. Appropriate devices for these purposes are pH-meters, available commercially as different models, e.g. stationary or portable devices. Changes of electromotive force of the cell can be ascribed (due to constant value of the reference electrode) to changes of the indicator electrode potential. The dependence of the potential,  $E$ , on ions concentration is described by Nikolsky equation, derived from classical Nernst equation:

$$E = E^0 + (2,3RT/zF) \log (c_i + K_{ij} c_j)$$

where  $c_i$  is the concentration of primary ions, for which the electrode is „sensitive”,  $c_j$  is the concentration of interferent ions (in Nikolsky equation the same charge,  $z$ , of primary and interferent ions is assumed). In a strict description concentrations should be replaced by ions activities<sup>1</sup>, however, in the case of dilute solutions insertion of concentrations to the equation does not result in a significant error.

<sup>1</sup> Activity is a product of concentration and activity coefficient:  $c_i f_i$ . For dilute electrolyte solutions, according to Debye-Hückel theory:  $-\log f_i = 0,5z_i^2 I^{1/2} / (1 + I^{1/2})$ , where  $z_i$  is the ion charge,  $I$  is the ionic strength,  $I = 0,5 \sum c_i z_i^2$ .

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$K_{ij}$  is the selectivity coefficient, representing influence of interferent ions on the potential (the selectivity coefficient should be possibly low, not higher than  $10^{-3} - 10^{-4}$ ),  $E^0$  is the constant related to the electrode,  $R$  is the gas constant,  $T$  is the temperature in K,  $F$  – the Faraday's constant.

The electrode potential is dependent on the concentration of “free”, i.e. hydrated ions, not forming complexes or deposits. The above given Nikolsky equation shows also that the sensitivity of an ISE, understood as the ratio of the signal change to corresponding concentration change is not high. For ions with +/- 1 charge (e.g. potassium cations or chloride anions) reducing concentration to 50% of its initial value results in theoretical electrode potential decrease equal to 18 mV (at 25°C). For instance, such concentration change may relate to physiological concentration of potassium ions in blood (from 3 to 6 mM). For ions of +2 charge, e.g. for calcium cations, the corresponding potential change is only 9 mV. However, in many cases even this low sensitivity is sufficient and resulting problems are compensated by measurement simplicity, low cost and no need of special pretreatment of the original samples.

## Classical solutions

Although construction and composition of components of typical ion-selective electrodes can be different, some features are common (Fig. 1). A typical electrode contains a membrane (solid – glass, crystalline or plastic – polymeric) separating the sample solution from the internal solution containing the same ions as those inducing sensitivity of the ISE (primary ions). Additional component is an internal electrode, immersed in the internal solution, this electrode is usually a silver wire coated by sparingly soluble silver chloride – silver / silver chloride electrode, connected to a cable.

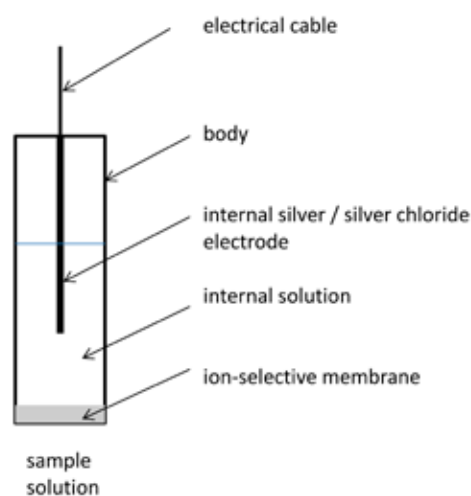


Fig. 1. Scheme of a typical ion-selective electrode

In case of glass electrode the membrane is a thin glass layer, of thickness close to 50 – 100  $\mu\text{m}$ , which can exchange hydrogen ions with the solution. The inner part of the sensor is usually filled

with hydrochloric acid solution (in aqueous solution fully dissociated to hydrogen and chloride ions) and a silver / silver chloride electrode is immersed in this solution. In this system the potential of the glass electrode is determined by potentials at all interfaces. This potential sum contains also the potential of the internal silver / silver chloride electrode, dependent on the chloride ions concentration in the internal solution. However, because this concentration is constant, the resulting potential is also constant. The next component of the potential sum is the membrane potential, linearly dependent on the ratio of hydrogen ions concentration in the sample solution and in the internal solution (where this concentration is constant). Therefore, the potential of the glass electrode is linearly dependent on the logarithm of hydrogen ions concentration, i.e. a linear dependence potential vs. pH is observed.

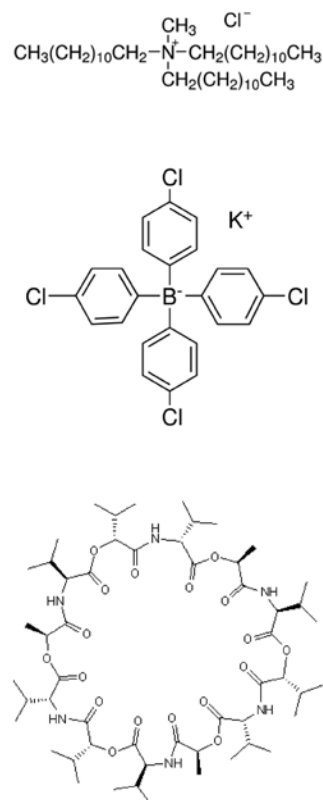
For a long time only the glass electrode has been ion-selective electrode of practical significance. However, in 1960ties new ideas appeared. In this group of sensors an electrode sensitive to fluoride ions, proposed by Frant and Ross, became very useful and was commercially available. Significance of this sensor kind is particularly high due to difficulties in fluoride ions assessment using other methods. In this electrode, in contrast to glass electrode, monocrystalline lanthanum(III) fluoride membrane is used, doped by europium(II) fluoride to enhance conductivity. This sensor is characterized by a linear dependence of the potential on the logarithm of fluoride ions concentration within the range from 1 to  $10^6$  M and by very high selectivity. The most significant interference are  $\text{OH}^-$  ions, thus the optimal pH range is 5–5.5, maintained by addition of acetic buffer (the solution must not be too acidic due to protonation of fluoride ions). The fluoride sensitive ISE was applied, e.g. do determination of fluoride ions added as a preservative to blood samples.

The next breakthrough, also observed in 1960ties, concerned application of ionophores – natural or synthetic organic ligands to obtain highly selective ISEs. Many synthetic ionophores were synthesized at ETH in Zurich, in the group of Simon. On the other hand, in 1970ties a new kind of membrane material was proposed – plastic, poly(vinyl chloride) (PVC) based membranes [2, 3]. These membranes can be prepared in a simple way – by preparing a solution of all components (with PVC) in a volatile organic solvent, typically tetrahydrofuran. Then, the volatile solvent is evaporated from this solution (this solution is called also a „cocktail“) and finally a membrane of thickness 100 – 200  $\mu\text{m}$  is obtained. This method is very useful due to its simplicity and stability of the produced membranes as well as easy composition tailoring. PVC based membranes found applications in many types of ISEs and became a “standard” in ion-selective electrodes construction, sensitive to e.g.  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{NH}_4^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ag}^+$  ions.

In a „classical” ion-selective electrode the PVC based membrane separates the sample solution from the internal solution, usually containing the primary ions. The membrane composition is optimized from the point of view of analytical parameters as well as mechanical and electrical properties. Such membrane contains typically 30% (m/m) PVC, around 65% of a plasticizer and (a few %) of ion-exchanger and ionophore. The plasticizer, typically di(2-ethylhexyl sebacate) or o-nitrophenyloctyl ether, is an organic solvent, immiscible with water and responsible for the membrane plasticity because PVC only is too hard and fragile. The ion-exchanger is a salt, where one of the ions (cation or anion) is a large and bulky organic ion (Fig. 2), characterized by high lipophilicity and therefore it is not released to the aqueous solution, neither to the sample solution nor to the internal solution. A counter-ion of the ion-exchanger is a simple hydrophilic ion (usually  $\text{Na}^+$ ,  $\text{K}^+$  or  $\text{Cl}^-$ ). Cation-sensitive electrodes contain a cation-exchanger in the membrane, i.e. a salt with organic anion, anion-sensitive electrodes contain an anion-

exchanger (a salt with organic cation). The ion-exchanger plays a double role: (i) it maintains electroneutrality of the membrane, when incorporated cations are bound with ionophore, the ion-exchanger releases mobile cations, (ii) the ion-exchanger presence increases the electrical (ionic) conductivity of the membrane. However, even in the presence of ion-exchanger, the resistance of the membrane is quite high, around  $10^5$ , or even  $10^6 \Omega$ .

The ionophore, as already mentioned, is an organic ligand which, particularly in organic lipophilic medium, strongly interacts with primary ions and therefore maintains high selectivity. A typical example of ionophore is natural ionophore, valinomycin, selectively interacting with potassium ions (Fig. 2).



**Fig. 2. Structures (from top) of: anion-exchanger (tridodecylomethylammonium chloride), cation-exchanger (potassium tetrakis(4-chlorophenyl) borate) and ionophore (valinomycin)**

The well-known ionophores interact mainly with cations. However, availability of ionophores for anions is very limited and in this case the factor determining the selectivity is affinity to the organic medium of the membrane, i.e. anion lipophilicity. Examples of highly lipophilic anions are  $\text{ClO}_4^-$  or  $\text{NO}_3^-$  ions, while lipophilicity of e.g.  $\text{SO}_4^{2-}$  or  $\text{F}^-$  anions is rather low.

### New ideas

The „classical” ion-selective electrodes with internal solution are characterized by very good analytical parameters – linear response range, usually from 1 to  $10^{-5}$  ( $10^{-6}$ ) M, stability of the recorded potential, short response time and high selectivity. However, a significant drawback of these sensors is the presence of the internal solution – this requires vertical position of the electrode to avoid leakage, the solution needs refilling, moreover, there are problems with miniaturization and shape tailoring. Thus, it would be reasonable to construct an internal solution – free sensor. The simplest idea, proposed in early 1970ties, was covering a solid metal (platinum or gold) or carbon electrode with ion-selective membrane. However, this construction simplification resulted in lower stability and poor reproducibility of the potential, caused by direct contact of two phases of quite different properties.

The membrane exhibits low ionic conductivity while the electrode support is a good electronic conductor. Since both phases have no common charge carriers, a „blocked” interface of low electrical capacitance is formed. Therefore, even flow of a very small current, typical for voltage measurements, could result in significant potential changes. The reason of observed instability can be also formation of a thin electrolyte solution layer between the support and the membrane with composition changing in course of the measurement.

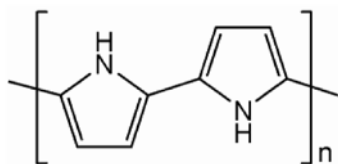


Fig. 3. Structure of polypyrrole

Significant progress, concerning construction and analytical parameters of ISEs could be noticed in 1990ties. A new idea concerning solution-free sensors was proposed, based on inclusion of additional layer between the membrane and support, called a solid contact [4]. The material applied as the solid contact was a conducting polymer, polypyrrole (Fig. 3), which due to presence of conjugate double bonds, after oxidation (and generation of positively charged carriers) is a quite good electronic conductor. Moreover, because the positive charge in the polymer structure is compensated by anions, this material is also ionically conducting. Thus, polypyrrole is mixed ionic / electronic conductor which can exchange ions with the membrane and electrons with the electrode support (Fig. 4). Due to possibility of charge exchange at all interface, there is no “blocking” and this ISE (called all-solid-state ISE) is characterized by similar analytical parameters as a “classical” ISE with internal solution. Similarly good results were observed after application of other conducting polymers.

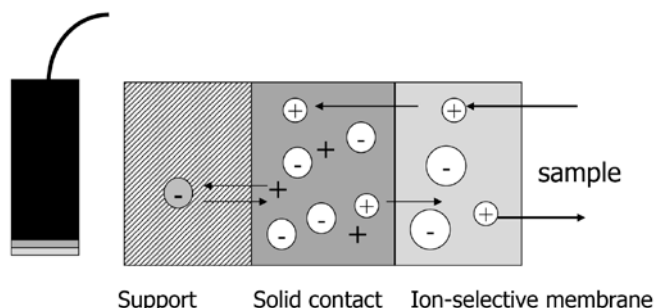


Fig. 4. Construction of all-solid-state ion-selective electrode (with solid contact) (left) and scheme of charge transfer at interfaces of such sensor (right)

In following years one could observe efforts to obtain cheap and disposable ion-selective electrodes. Some new ideas were proposed, typical for semiconductor technology, concerning flat sensors production by deposition of ion-selective membranes on silicon support [3, 5], other authors implemented screen-printing method to produce electrodes [6, 7]. We also proposed two novel ideas of disposable ISEs. One of them concerns cheap disposable plastic electrodes, where a foil support is covered by a conducting polymer dispersion [8]. After dispersing agent (e.g. water) evaporation one obtains a conducting polymer layer, playing a double role – of electronically conducting substrate and solid contact. This layer is then covered by an ion-selective membrane. The second kind of electrodes (described in our paper published in 2014) is all-solid-state ISE, where all component layers are deposited on a foil using spray-coating method. In both cases the sensors were characterized by similar and good analytical parameters.

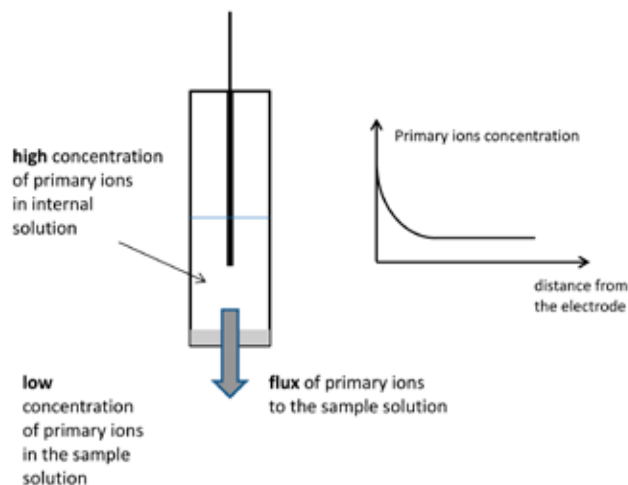


Fig. 5. Scheme showing the reason of elevated detection limit for ion-selective electrode

The end of 20<sup>th</sup> century has brought a significant breakthrough in understanding mechanisms of ion-selective electrodes operation. This progress resulted in methods of significant detection limit lowering (to  $10^{-9}$ , or sometimes even  $10^{-12}$  M, what is typical for much more expensive spectroscopic methods). One of leading contributors to this progress was Tomasz Sokalski (Ph.D.) from University of Warsaw (in collaboration with group of Prof. Pretsch from ETH Zurich), who unraveled the significant role of ion fluxes under currentless (potentiometric) conditions [10, 11]. If an ISE is immersed in solution of very low primary ions concentration, a spontaneous flux of these ions from the internal solution (where the concentration of primary ions is usually high), across the membrane to the sample solution occurs (Fig. 5). Thus, the primary ions concentration in the solution layer adjacent to the membrane is higher than in the solution bulk, this effect results in artificially enhanced signal and is origin of the detection limit close to  $10^{-5} - 10^{-6}$  M. A solution was proposed, based on “buffering” of primary ions in the internal solution, e.g. by adding complexing ligands interacting with primary ions and resulting in significant decrease of their concentration in the internal solution.

New ideas concerned also the material of ion-selective membranes. Some papers appeared, proposing to replace PVC by polyacrylates [12]. Polyacrylates are beneficial due to variety of monomers, enabling easier tailoring of the membrane properties. These membranes can be also easily produced by photopolymerization, directly on the electrode support, moreover, in some cases, these materials do not require plasticizer addition. This is a significant advantage due to possible release of the plasticizer, e.g. from classical PVC based membranes. Additional advantage of polyacrylates is lower mobility of ions in such membrane, compared to PVC. Although this results in membrane resistance increase, lower mobility of ions reduces the ionic fluxes magnitude, what (as described above) is advantageous from the point of view of detection limit lowering.

In recent years there has been great interest in nanostructures and nanotechnology, in different areas of science and technology. Nanoparticles and generally nanostructured materials can be also used as components of ISEs. They can be additives to membranes, e.g. quite heavy (in molecular scale) gold nanoparticles covalently connected with ionophore molecules can serve as a kind of anchor, immobilizing ionophore in the membrane [13]. Another example concerns addition of platinum nanoparticles to a polyacrylate membrane resulting in significant decrease of its resistance [14]. Therefore, the membrane is still characterized by low ionic mobility (advantageous for detection limit lowering), but on the other hand lower resistance results in better stability of the recorded potential.

Due to high surface area / volume ratio of noble metals or carbon (e.g. carbon nanotubes) nanoparticles, the nanoparticles layer / electrolyte solution interface is characterized by a high electrical capacitance. Therefore such nanostructural materials can be an advantageous alternative to typical conducting polymer based solid contacts. Also in our idea of electrosprayed ISEs [9] carbon nanotubes played a double role of electrode support and a solid contact.

Additional possibility of nanostructures application is modification of e.g. gold nanoparticles or graphene by appropriate ligand interacting with selected metal cations. In this way low resistance ion-selective membranes can be obtained of promising analytical parameters [15].

### Galvanostatic polarization of ion-selective electrodes

Ion-selective electrodes have been usually applied in potentiometry, i.e. under currentless conditions. However, since beginning of the 21<sup>st</sup> century typical electrochemical current based techniques have been applied to study and utilize ISEs, among these method the most popular is galvanostatic polarization. Although the measurements protocol becomes more complicated under such conditions and the apparatus costs are higher than for typical potentiometry, there are significant benefits. Initially, the galvanostatic polarization was applied to decrease the detection limit. Assuming that the detection limit is affected by spontaneous flux of primary ions from the internal solution, across the membrane, to the sample solution, application of appropriate cathodic current can induce ionic flux in the opposite direction, compensate the spontaneous leakage and ultimately decrease the detection limit [16]. Unfortunately, a weak point of this method is difficulty in forecasting the optimal current, which moreover can be dependent on sample composition.

Later, other ideas were proposed. Galvanostatic polarization, in the form of current pulses, proved to be useful in determination of organic ions (on example of multicharged ionic forms of heparin and protamine, controlling the process of blood coagulation) [17]. For such ions of high charge the Nikolsky equation predicts very low slope of equilibrium potential vs. logarithm of analyte concentration dependence, thus, ISEs are in this case useless. However, the experimentally observed slopes of potentiometric dependences for such ions are quite high, since organic ions (of high lipophilicity) are spontaneously extracted into the membrane. This process is irreversible and thus the recorded potential and potentiometric calibration plot is time dependent and irreproducible. In this case pulse galvanostatic polarization was very useful, by controlled forcing ion flow, thus maintaining a stable analytical characteristic with high slope of potential vs. logarithm of concentration dependence.

Galvanostatic polarization can be also used to elevate the theoretical limit of potentiometric method sensitivity, represented by the slope of linear dependence of potential on logarithm of concentration, not exceeding 59 mV for ions of +/- 1 charge at 25°C. Application of cathodic polarization can result in controlled decrease of cations concentration in solution layer close to the electrode (compared to solution bulk) and in this way in reproducible super-Nernstian slope, higher than theoretical one.

Galvanostatic polarization can be used not only to affect the detection limit or sensitivity. It can be applied to controlled change of membrane composition and thus to improvement of analytical parameters under open-circuit (currentless) conditions. It was generally believed (and appropriate protocols were used) that stable optimal potentiometric characteristics could be obtained for fully saturated ion-selective membrane. Full saturation means in this case that the charge of ion-exchange sites (total charge of lipophilic ions of the ion-exchanger in the membrane) is compensated by primary

ions. However, in this case release of primary ions to the solution is likely and the detection limit can be higher. It has been proved that better (lower) detection limit can be obtained for not fully saturated membrane, i.e. the membrane contains not only primary but also other ions of the same charge sign. A convenient method of ion amount adjustment by precise incorporation of ions to the membrane can be again galvanostatic polarization [18].

The above presented applications were only a few examples of possibilities offered by electrochemical techniques in the field of ISEs. Research in this area has been intensively developed recently and is object of interest of many research groups worldwide. Studies concerning this group of sensors are also carried out in Poland, e.g. at Warsaw University of Technology, University of Warsaw, AGH University of Science and Technology and Gdansk University of Technology.

### Summary

Ion-selective electrodes are established and commercialized tool of chemical analysis. These sensors have been used for many years, in their over 100-years history some breakthrough ideas have appeared: application of ionophores, plastic membranes, detection limit lowering, internal solution-free constructions, new polymeric membrane materials, applications of nanostructured materials, current based methods. It seems that these sensors will be still routinely used in various areas, but on the other hand, this field is still open for scientific research. Development of other branches of chemistry as well as progress of electronics and nanotechnology can give rise to impact on ion-selective sensors development.

### Acknowledgement

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## Aktualności z firm

### News from the Companies

Dokończenie ze strony 377

#### Nowy Prezes Zarządu i Dyrektor Generalny Basell Orlen Polyolefins

Joseph Tanner z LyondellBasell został powołany przez Basell Europe Holdings B.V. na stanowisko Prezesa Zarządu i Dyrektora Generalnego Basell Orlen Polyolefins Sp. z o.o. Z dniem 15 czerwca 2015 r. zastąpi on Thomasa Srnkę, który zarządza Basell Orlen Polyolefins od marca ub. r. Joseph Tanner związany jest z LyondellBasell od ponad 20 lat i do czasu objęcia nowego stanowiska, pełni funkcję Dyrektora Dywizji Acetylów, Butanodiolu, Tlenku Etylenu/Glikolu Etylenowego w LyondellBasell. Doświadczenie zdobywał m.in. w obszarze produkcji, badań, sprzedaży i finansów, które w znacznej mierze związane były z biznesem polimerowym. Pełnił wiele kierowniczych funkcji. Joseph Tanner studiował na Uniwersytecie Purdue w USA, gdzie zdobył tytuł inżyniera mechaniki. Pochodzi ze Stanów Zjednoczonych. (kk)

(<http://www.basellorlen.pl>, 9.06.2015)

#### Powołano Zarząd Grupy LOTOS SA

Rada Nadzorcza Grupy LOTOS SA do składu Zarządu Grupy LOTOS SA IX wspólnej kadencji powołała wszystkich dotychczasowych członków zarządu. VIII kadencja Zarządu zakończy się w dniu 28 czerwca 2015 r. IX wspólna kadencja Zarządu rozpocznie się z dniem 29 czerwca br.

Pan Mariusz Machajewski obejmie funkcję Wiceprezesa Zarządu ds. Ekonomiczno-Finansowych. Pan Zbigniew Paszkowicz obejmie funkcję Wiceprezesa Zarządu ds. Poszukiwań i Wydobywania. Pan Marek Sokółowski obejmie funkcję Wiceprezesa Zarządu ds. Strategii i Rozwoju. Pan Maciej Szozda obejmie funkcję Wiceprezesa Zarządu ds. Produkcji i Handlu. 29 maja br. Rada Nadzorcza powołała Pana Pawła Olechnowicza na funkcję Prezesa Zarządu. Pan Paweł Olechnowicz funkcję Prezesa Zarządu Grupy LOTOS SA pełni od 2002 r. Funkcję Wiceprezesa Zarządu Grupy LOTOS SA pełni nieprzerwanie od 2006 r. Pan Mariusz Machajewski, wchodząc w skład Zarządu VI, VII oraz VIII kadencji. Związany z Grupą LOTOS SA (wcześniej Rafineria Gdańska SA) od 1997 r.

Funkcję Wiceprezesa Zarządu Grupy LOTOS SA pełni od 2012 r. pełni Pan Zbigniew Paszkowicz – powołany przez Radę Nadzorczą Spółki do składu Zarządu VIII kadencji. Z Grupą LOTOS SA (wcześniej Rafineria Gdańska SA) związany jest zawodowo od 1992 r.

Funkcję Wiceprezesa Zarządu Grupy LOTOS SA pełni nieprzerwanie od 2002 r. Pan Marek Paweł Sokółowski wchodząc w skład Zarządu IV, V, VI, VII oraz VIII kadencji. Z Grupą LOTOS SA (wcześniej Rafineria Gdańska SA) związany jest zawodowo od 1973 r. Funkcję Wiceprezesa Zarządu Grupy LOTOS SA pełni od 2009 r. Pan Maciej Szozda wchodząc w skład Zarządu VII oraz VIII kadencji. (abc)

(Informacja Grupa LOTOS, 26 czerwca 2015 r.)

#### NOWE PRODUKTY

##### Pigmenty efektowe Lumina® Royal

Obecnie, w ramach serii produktów Lumina® Royal, firma BASF oferuje pięć pigmentów do zastosowań w produkcji tworzyw sztucznych. Pozwala to producentom tworzyw sztucznych na całym świecie na tworzenie atrakcyjnych projektów w odcieniach koloru niebieskiego i koloru miedzi. Już w 2010 r. firma BASF wprowadziła na rynek pigment efektowy Lumina® Royal Blue do tworzyw sztucznych. Pigmenty Aqua, Indigo, Magenta i Copper zostały zaprezentowane na CHINAPLAS – międzynarodowych targach przemysłu tworzyw sztucznych i gumy, które odbyły się w Guangzhou w Chinach, w dniach 20–23 maja 2015. Podczas seminarium firma BASF po raz pierwszy zaprezentowała kompletną serię dla klientów z branży tworzyw sztucznych.

Pigmenty z serii produktów Lumina® Royal charakteryzują się wysokim połyskiem, czystością i chromatycznością. Pigmenty Blue, Aqua, Indigo i Magenta pokrywają zielono- i czerwono-niebieskie widmo kolorów, natomiast Lumina Royal Copper oferuje efekt nieprzezroczystego jasnego odcienia miedzi. (kk)

(<http://www.basf.pl>, 25.05.2015)

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