

Ionogels – materials containing immobilized ionic liquids^{*)}

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Abstract: Ionic liquids (IL) are characterized by unique properties which are the reason for the continuous growth of interest in these compounds for a large variety of applications. However, in many applications there is a need for immobilization of IL within solid matrices while maintaining IL specific properties. Such stable solid-liquid materials are called ionogels. This review provides basic information about ionogels, their preparation, properties and application with an emphasis on ionogels with polymer matrices.

Keywords: ionic liquids, ionogel, classification of ionogels, properties of ionogels, application of ionogels.

Jonożele – materiały zawierające immobilizowane cieczce jonowe

Streszczenie: Duże zainteresowanie cieczkami jonowymi (IL) wynika z ich wyjątkowych właściwości. Wiele zastosowań IL wymaga immobilizacji IL w stałych matrycach, ale z zachowaniem specyficznych właściwości tych cieczy. Takie stabilne materiały, stanowiące układy ciecz-ciało stałe, nazywane są jonożelami. Niniejszy artykuł stanowi przegląd literatury dotyczącej klasyfikacji, metod otrzymywania i właściwości, a także możliwości zastosowań tych materiałów. Szczególną uwagę poświęcono jonożelom, których matryce stanowią polimery.

Słowa kluczowe: cieczce jonowe, jonożel, klasyfikacja jonożeli, właściwości jonożeli, zastosowanie jonożeli.

The concept of ionogel is inextricably associated with the concept of ionic liquids (IL). The latter are the subject of interest of scientists and engineers for many decades, owing to their peculiar and tunable properties compared to non-ionic compounds, including a wide liquid range, high ionic conductivity (in the range of 0.1–20 mS/cm [1]), negligible vapor pressure, and high electrochemical (up to about 5 V [1, 2]) and thermal stability (the onset of thermal decomposition most often is > 200 °C [3]), as well as a significant ability to solubilize organic, inorganic, and polymeric materials.

Ionic liquids (IL) are organic salts with melting temperature below 100 °C. When the melting temperature is lower than 20 °C, they are called room temperature ionic liquids (RTIL).

IL consist of only ions [the most popular cations and anions are presented by formulas (I)–(XI)] and therefore their properties differ significantly from non-ionic (“molecular”) liquids. The chemical nature of IL is determined

by their structure and by the interplay of interionic interactions such as Coulombic forces, van der Waals inductive and dispersion interactions, hydrogen bonds, π - π and n - π interactions, *etc.* The main directions in investigation of IL are their applications as [4, 5]:

- alternative green, designable solvents and media to replace traditional volatile organic solvents in clean technologies such as organic reactions, catalysis, extraction, and separation;
- safe electrolytes for electrochemistry and a number of energy-related applications, especially in batteries, chemical sensors, fuel cells, supercapacitors, and dye-sensitized solar cells.

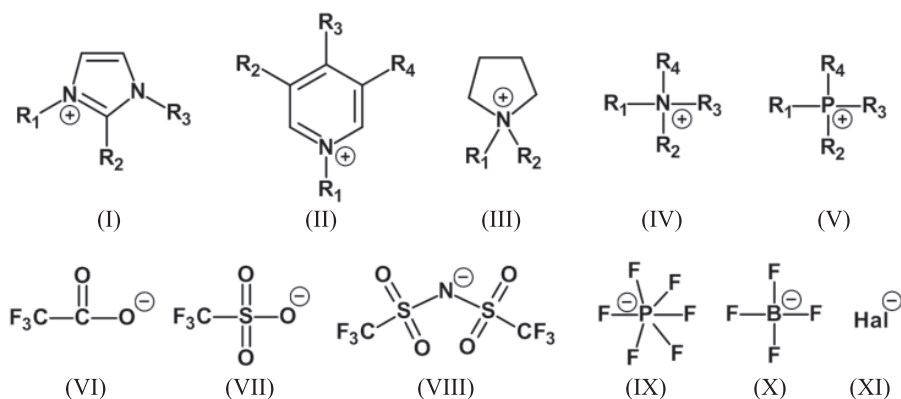
In many applications IL serve as device components. However, to be applied in a device, IL must be immobilized on a solid carrier or solid matrix, because it is liquid by definition. The matrix should immobilize IL without changing its unique properties needed for the device to work [6]. Due to immobilization of a liquid IL in a solid matrix we obtain a gel material, which has been termed ionogel or ion-gel [7, 8] (gel within which the liquid component is made up entirely of ions [9]). Therefore, ionogel can be considered as a material consisting of a solid matrix (which can be organic or inorganic) and IL entrapped in it.

This short review presents the basic information on ionogels, their preparation, properties and applications, focusing mainly on ionogels with polymer matrices.

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GENERAL CHARACTERIZATION OF IONOGENS

Ionogels combine the physical properties of both the polymer and the physically entrapped IL [10] keeping the main properties of IL (ionic conductivity, non-volatility, non-flammability) except outflow, and the mechanical stability of the host matrix. The matrix content in ionogels can be very low (even only 2–3 % in the case of silica matrix [11, 12], and of the order of 10 % with polymer matrices [13]). Depending upon the chemical composition and other factors, ionogels vary in chemistry from fairly rigid solid to soft material [14].

In some papers [6, 7, 10, 15, 16] ionogels are considered as hybrid materials, in which the properties of IL are hybridized with those of another component. This relies on the intimate combination of IL and the solid-like network. These novel hybrid materials are indicated for possible applications as catalytic, separation, or ion conducting membranes. There are two main approaches to the immobilization of IL [6, 17, 18]:

- by chemical attachment of ionic species to a solid support,
- by the physical fixation of IL (without covalent linkage, *e.g.*, swelling) within a matrix.

However, it is believed that with the first approach, although interesting for, *e.g.*, catalysis applications, the materials cannot be regarded as ionogels as no liquid phase is concerned (they contain ionic complexes which show strong similarities to ionic liquids) [15, 19, 20].

Although it is commonly accepted that the ionogels consist of solid matrices and IL, sometimes authors [21] consider as ionogels (with polymer matrix) only those materials that have full compatibility between the matrix and IL, *i.e.*, materials which show a single temperature of glass transition (T_g). Other works, *e.g.*, review [14], define ionogels as materials containing IL confined in nanopores, independently on the matrix type; while in other works the term ionogel includes all materials containing both forms of IL, *e.g.* refs. [6, 7, 16].

Another controversy is associated with widely recently studied poly(ionic liquids) (PIL). These polymers are capable of acting as molecular gelators for organic solvents or water, which form organogels [22] or hydrogels [23]. In such cases the PIL forms the solid matrix whereas the liq-

uid phase is water or an organic solvent; this is in contradiction with the definition of ionogels as materials containing IL. When PIL are used as matrices for IL [24–26] then the resulting materials are true ionogels. However, in some reports PIL are considered as ionogels [16].

It is worth to mention that compared to hydrogels or organogels, ionogels show excellent mechanical properties such as higher mechanical strength, tunable elastic response and rapid recovery ability after the deformation [27].

CLASSIFICATION OF IONOGENS

Ionogels can be classified according to various criteria [9]. The most popular is the classification by the matrix type [7, 14]:

- inorganic ionogels (with matrices formed by oxide nanoparticles, carbon nanotubes or oxide networks synthesized by sol-gel process);
- organic-inorganic ionogels (based on polymer matrices containing inorganic additives);
- organic ionogels (prepared using low molecular weight gelators or polymer matrices).

Another important type of ionogel classification is their assignment to physical or chemical gels, depending on the type of matrix formation. In physical gels, the formation of the three-dimensional network occurs due to weak and reversible physical interactions, whereas in chemical gels the matrix is formed by covalent bonding.

Classification of ionogels according to these criteria is schematically shown in Fig. 1.

PREPARATION OF IONOGENS

Inorganic ionogels

Inorganic ionogels are usually prepared by dispersion of inorganic particles in ionic liquids or using the sol-gel technique. Pristine single-walled carbon nanotubes formed gels after being ground when mixed with imidazolium RTIL [28] or by heating and ultrasonication in the presence of IL [29]. The formation of the dispersion was possible due to cation- π or electrostatic interaction between IL and carbon nanotubes [30]. However, silica

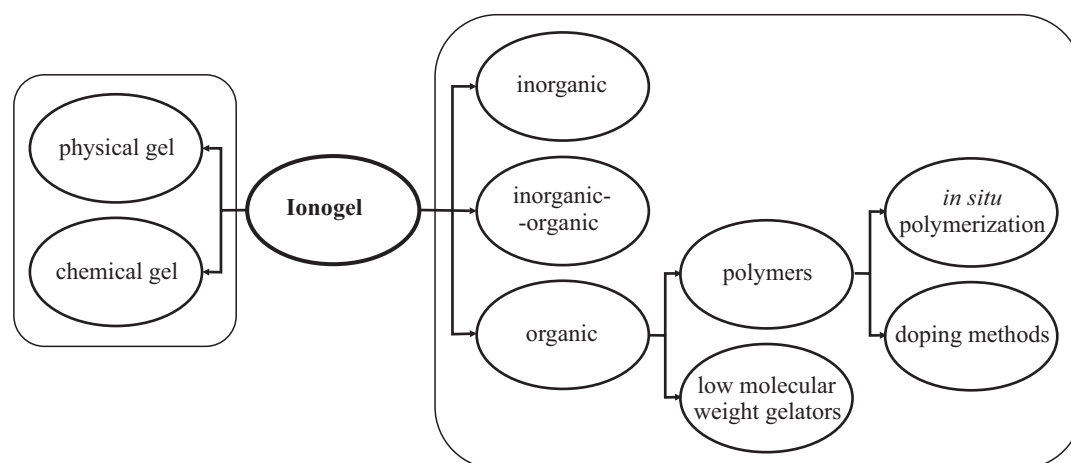


Fig. 1. Classification of ionogels

dispersions in IL appeared to be unstable due to colloidal aggregation of silica particles which was explained by extremely high ionic strength and resulting surface-charge screening [31].

Therefore, the best way became the sol-gel process leading to formation of nano-porous oxide matrices in which IL in the form of interconnected networks get entrapped. Two sol-gel routes were used: hydrolytic [12, 32] and non-hydrolytic [33–35]. In the case of silica matrix, the precursors are silicon alkoxides (tetramethylorthosilicate and tetraethylorthosilicate). In the hydrolytic route, the hydrolysis, to start the formation of gelling network like $-\text{Si}-\text{O}-\text{H}-$, can be carried out by water or by alcohol. In the non-hydrolytic route, the gel-catalyzing agents (instead of water/alcohol) are formic acid, glycolic acid, *etc.* [14].

Organic-inorganic ionogels

This type of ionogels can be prepared by mixing of a polymer solution in IL with inorganic oxide nanofiller without [36] or with the use of a volatile solvent [24], or by *in situ* formation of inorganic filler in the polymer/IL solution in the non-aqueous sol-gel route [37]. Introduction of inorganic filler into IL/polymer gel can help also to reduce the often observed plasticizing effect of IL on the polymer matrix. On the other hand, IL can act as a compatibilizer between the polymer matrix and the nanofiller [7, 38].

Organic ionogels

Organic ionogels can be prepared using low molecular weight or high molecular weight (polymer) gelators.

Gelation of IL with the use of low molecular weight organogelators (LMWOG)

Addition of LMWOG to IL at elevated temperatures causes physical gelation upon cooling. Gelation occurs due to hydrogen bonding, $\pi-\pi$ interactions or electrostatic interactions. As LMWOG carbohydrates [39–41], amino acids/gluconic acid hydrophobic derivatives [42] or others [43] were used.

Gelation of IL by organic polymers (polymer ionogels)

The polymer-based ionogels can be obtained in two main ways [13]: doping methods (way A) and polymerization methods (way B).

Doping methods include:

- the solution casting method (solvent blending), which involves dissolution of a polymer and IL in a volatile solvent; the homogeneous mixture is then casted and dried to remove the solvent [44, 45];
- the soaking method (impregnation), which consists of swelling of a polymer in IL [46, 47] or by formation of microporous polymer membranes in multistage procedures, followed by soaking the so obtained membranes with IL.

The polymerization method is an *in situ* process based on the polymerization or crosslinking of monomers dissolved in IL [15]. Both chain as well as step polymerization processes have been used.

Polymer materials applied in the solution casting method (way A) include homopolymers, copolymers and hydrocarbon polymers or block copolymers. This method enables mixing polymers and IL in various proportions, however the resulting materials have often poor mechanical properties which need to be further improved by crosslinking [48]. Moreover, it is not possible to use directly crosslinked polymer matrices.

In the soaking method (way A) at first the polymer membrane is prepared by direct casting of homopolymer, copolymer, and block copolymer or polymer blend solutions to form dense or porous membranes which are subsequently soaked in a solution of IL [13, 49]. Impregnation is the simplest way to prepare polymer-based ionogels. This method enables application of crosslinked polymer matrices, however, it does not allow for the exact control of the amount of IL absorbed because the composition range is limited by the maximum swelling capacity of the polymer membrane. The process of swelling results from the balance between repulsive and attractive phenomena: the expansion of the polymer takes place due to the entropic diffusion but swelling is counter-acted by

elastic forces within the polymer chain and inter-chain attractive forces; the process is controlled by Gibbs energy of mixing [47].

Polymers which were most often used as polymer matrices in doping methods (way A) were fluoropolymers – mainly poly(vinylidene fluoride) (PVDF) and its copolymers, *e.g.*, poly(vinylidene fluoride-*co*-hexafluoropropylene) (solvent casting) [50, 51] or Nafion™ [51]; other polymers include sulphonated poly(ether ether ketones) and sulphonated polyimides [52]. Doping methods may include also preparation of ionogels from natural polymers, like chitin [53], its mixture with cellulose [54] or gelatin [16, 55].

Way B, *i.e.*, polymerization of monomers in IL, has been widely used for the preparation of ionogels. Common vinyl monomers are soluble in common IL and can be polymerized *via* free radical polymerization as well as in other polymerization processes [56–58]. However, ionogels tested for specific applications were most often prepared using radical initiators. Free radical polymerization can be initiated chemically (with the use of thermal initiators) and photochemically (with the use of photo-initiators).

Preparation of ionogels (especially in the form of membranes) by the polymerization induced by thermal initiators has several drawbacks: it needs relatively high temperatures (70–80 °C) and long reaction times (2–24 h) [59–65]. Special advantages offers the light-induced process: short polymerization time (seconds to minutes), low reaction temperature (room or ambient), full control of the process (the polymerization starts when the light is switched on and stops when the light is turned off), and spatial resolution [15, 66–74].

There are also reports on preparation of ionogels by polyaddition reaction in IL (multifunctional epoxy compounds with amines) [75, 76] and very recently by thiol-ene process [77, 78]; when the latter is induced photochemically, it retains all the benefits associated with this type of initiation described above.

One of the latest papers reports on the preparation of HEMA-based ionogels in a deep eutectic solvent (DES) [79]; however, opinions are divided whether DES are ionic liquids [80] or not [81].

MORPHOLOGY AND PROPERTIES

When an ionogel has an inorganic matrix, it obviously forms a two-phase structure, where the solid and liquid phases percolate throughout each other. In the case of polymer matrices the situation is more complicated. Here the key point is the miscibility (compatibility) between the polymer and IL. In such a case the properties may be affected by the nanometer scale assembly.

Many popular polymers are hardly soluble in IL, although if a proper combination of the ions is selected, IL can be used as solvents for poorly soluble macromolecules [21]. Therefore, polymerization of monomers in IL or pre-

paration of ionogels by solvent casting method often leads to phase separated materials. The separated domains of IL can vary considerably in size, from nano- to micro-sized channels. Such a different morphology of IL/polymer materials can be reflected by their thermal properties: in the case of full compatibility of the components the ionogel shows only one glass transition temperature (T_g), lower than that of the neat matrix and higher than that of pure IL (IL serves as a plasticizer) [6]. In the case of phase separation two T_g values are observed (derived from the matrix and IL). However, for phase separated ionogels it is preferred to form two interpenetrated nanophases (induced for example by polymerization) and to fix them (by gelation) before the macroscopic phase separation takes place.

When compatible systems are needed, a suitable polymer matrix should be selected to hold IL effectively inside the polymer network by osmotic pressure. Therefore, polymer solubility (or compatibility) in IL has a major impact on the morphology and performance of the resulting ionogels [82]. In general, the solubility of homopolymers could be qualitatively explained by treating polymer/IL as a ternary system: polymer, cation and anion. As it was shown by recent studies on the solubility of poly(methyl methacrylate) (PMMA) in IL, the determining factor is the hydrophobicity of the anions, whereas the effect of the non-polarity of the cation can also contribute to the overall PMMA solubility [82, 83]. However, strong interactions between polymers and bulky cations in IL are relevant for higher dissolution performance [83].

Most studies on the combined use of IL with polymers described compatible systems; however, some of them used the phase changes or phase separation of a polymer in IL [21].

The two-phase structure can lead to interesting behavior of IL. When the dimensions of matrix pores are comparable to the size of IL, we can observe the so-called “confinement effect”. Due to spatial restriction and low dimensionality of the “confining matrix”, the physicochemical properties of IL, like phase transitions, molecular dynamics or thermodynamics, can be substantially modified by confinement in the nanopores of the host matrix [7, 14, 84]. The most known examples are silica ionogels. The behavior of phase transitions of these materials depended on the degree of confinement (controlled by IL/silica molar ratio). At the highest degrees of confinement (*i.e.*, the smallest space for IL) neither cold crystallization nor melting peak derived from IL were observed. At lower degree of confinement melting temperature (T_m) was lowered with respect to the pristine IL, whereas the cold-crystallization temperature was increased. In all the cases the glass transition was unchanged [7, 8, 15]. There was proposed an explanation that the change of the melting temperature occurs because the pore size changes the anion-cation distance [85]. Moreover, the chemical nature of the matrix (pore walls) and IL ions and resulting interactions have a strong impact on the thermal behavior of ionogels [7, 86]. In more detail the confinement effects are described in [14].

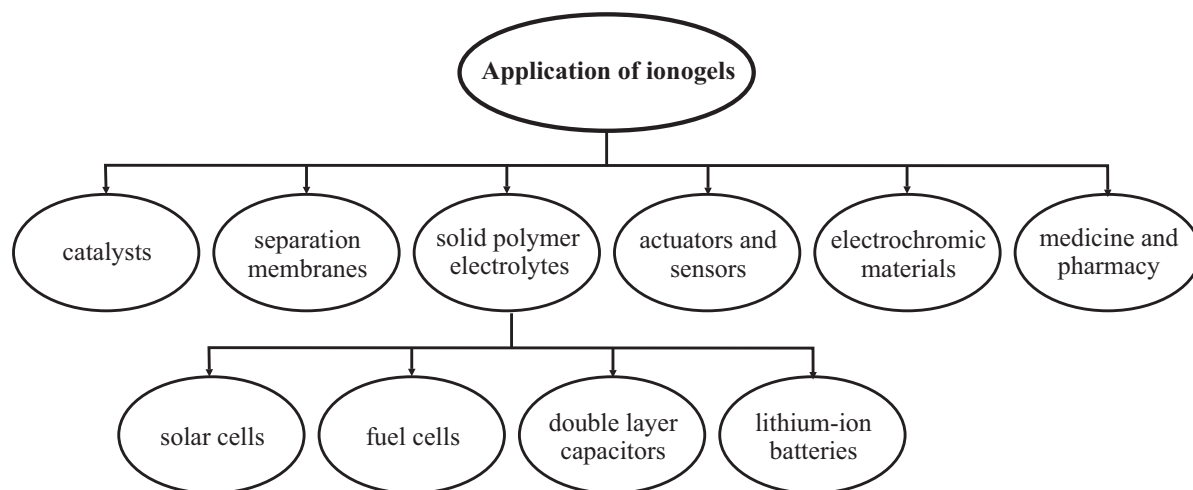


Fig. 2. Potential applications of ionogels

The effect of IL confinement was observed also in a modified polyacrylate-based matrix [87]. T_g and T_m values of IL increased due to confinement, whereas crystallization peak was not observed. Interesting observation was that the ionic conductivity in these gels was predominantly due to anions. For the materials based on epoxy resins the extent of confinement was defined by the amount of extractable IL. Materials confining IL (with IL content below 40 %) were insulating with a high Young's modulus, while those not confining IL (with IL content > 40 %) were ion conducting with a low Young's modulus. It was ascribed to the transition from the morphology in which IL forms discrete phases to that with continuous IL phase [76].

APPLICATIONS OF IONOGELES

As indicated above, ionogels can be used everywhere, where IL would find application in a solid form but preserving all the attributes of its liquid state. Some potential applications of ionogels are shown in Fig. 2.

A lot of attention has been paid to electrochemical applications. This is associated with the high conductivity of IL. However, immobilization of IL in matrices usually decreases its ionic conductivity. To be useful in electrochemical applications, an IL-containing material should have the conductivity ≥ 1 mS/cm. Because the conductivity of IL lies in the range of 0.1–20 mS/cm it is important to maintain a similar level of conductivity in ionogels [18].

Ionogel membranes for energy applications are usually applied as solid polymer electrolytes (SPE). In fact SPE are a type of gel electrolytes in which IL serve both as charge carriers and plasticizers [18].

Investigation of ionogels for energy application covers electrolytic (ion-conducting) membranes for electric double layer capacitors [21, 88–90], Li-ion batteries (SPE containing Li salts) [91–94], dye-sensitized solar cells (DSSC; a typical DSSC comprises a nanocrystalline semiconductor, *i.e.*, titanium dioxide, layer electrode, a sensi-

tizer adsorbed onto the semiconductor surface, an electrolyte with a dissolved iodide/triiodide redox couple, and a platinum counter-electrode) [95–99] and fuel cells (electrochemical devices able to convert chemical energy directly into electrical energy; they are based on proton-exchange membranes, containing protic IL) [100–102]. Ionogels were also used for formation of inkjet-printed microstructures [103].

IL are able to dissolve a wide range of substances, from organic compounds to metal salts and gases. This property has been used to apply ionogels in separation processes (*e.g.* for CO₂): the transport (permeation) rates and selectivity strongly depended on the nature of the supporting polymer membrane [104, 105].

IL in the form of ionogels were tested as catalysts. They provide facile nanoparticle entrapment, enhanced homogeneous catalyst performance, contamination free Brønsted acidity, and protective environments for biocatalysis [9]. Although most popular is application of silica matrices [7, 9, 106], ionogels based on polymer matrices were found to be very effective [107].

Another interesting application are actuators, which can be applied for example in intelligent robots, electrochemical sensors and biosensors. Their typical structure is composed of one ionically conductive electrolyte sandwiched between two electrically conductive electrodes, which can bend to allow the redistribution of different sizes of cations and anions under the applied voltage [10, 108–111].

It is also worth to mention application of appropriately doped ionogels as electrochromic materials (which change color upon the application of an applied electrical field) [112–114].

Still another possibility is application of ionogels for drug delivery systems in order to minimize drug degradation and loss, to prevent harmful side effects and to increase drug bioavailability [115–117] and as biocompatible biomaterials [118].

CONCLUSIONS

Ionogels are a new class of materials, still emerging, with a huge application potential. The appropriate choice of IL and the matrix with possibility of structuration on the nanometer scale can lead to novel materials with special properties and to targeted synthesis of ionogels which fit specific tasks. An additional advantage is reusability of IL, which gives a possibility of the recycling of IL immobilized in ionogels. All these make ionogels very promising candidates for the future use and the subject of many intensive investigations.

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60. Zjazd Polskiego Towarzystwa Chemicznego Wrocław, 17–21 września 2017 r.

Hasło Zjazdu brzmi: „Chemia w mieście spotkań”.

Wykłady i sesje posterowe będą się odbywały na Politechnice Wrocławskiej, Uniwersytecie Wrocławskim i na Uniwersytecie Przyrodniczym.

Zaproszenie do wygłoszenia wykładów przyjęli m.in.: prof. Guy Salvesen (Sanford Burnham Medical Research Institute, USA), prof. Ivan Huc (Bordeaux University, Francja), prof. Norbert Sewald (Bielefeld University, Niemcy), prof. Stefan Kaskal (Dresden Technical University, Niemcy).

Polskie Towarzystwo Chemiczne utworzone w 1919 r. zrzesza pracowników wyższych uczelni, nauczycieli, a także studentów i sympatyków tej dziedziny wiedzy. Jego głównym celem jest popieranie rozwoju nauk chemicznych i szerzenie wiedzy chemicznej wśród społeczeństwa.

Coroczny zjazd PTChem jest największą konferencją chemiczną organizowaną w Polsce, gromadzącą zwykle ok. 1000 osób. To wyjątkowe spotkanie chemików ma już długą tradycję – pierwsze 4 zjazdy odbyły się przed II wojną światową, a w 1948 r. to Wrocław gościł pierwszy powojenny Zjazd PTChem. Oddział Wrocławski organizował także zjazdy w latach 1967, 1979, 1998 i 2004. Po 13 latach przerwy po raz szósty będziemy gościć chemików z całej Polski podczas 60. Zjazdu – podwójnie jubileuszowego, bo przypadającego w roku 150-lecia urodzin Marii Skłodowskiej-Curie.

Uczestnicy konferencji będą mogli nie tylko wysłuchać interesujących wykładów i zaprezentować wyniki swoich najnowszych badań, ale również poczuć wyjątkową atmosferę naszego miasta. Wśród imprez towarzyszących zaplanowano m.in. koncerty w Narodowym Forum Muzyki i w Kościele Uniwersyteckim.

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