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Hybrid Materials – Past, Present and Future.

Abstract: Hybrid materials represent one of the most growing new material classes at the edge of technological innovations. Unique possibilities to create novel material properties by synergetic combination of inorganic and organic components on the molecular scale makes this materials class interesting for application-oriented research of chemists, physicists, and materials scientists. The modular approach for combination of properties by the selection of the best suited components opens new options for the generation of materials that are able to solve many technological problems. This review will show in selected examples how science and technological driven approaches can help to design better materials for future applications.

Keywords: Sol-gel, nanocomposites, porous materials, coatings biomaterials.

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1 Introduction

The synergistic combination of inorganic and organic compounds in one material to achieve improved properties is as old as mankind. Most of the times such material combinations were developed, because there was a lack of the required material characteristics in the traditional landscape of materials which could only be filled by merging two totally different single components. Applying this approach one can find many examples in the history of human beings. A very old example is the combination of straw –in principle an organic fiber - and clay – an inorganic matrix - for the construction of houses. This mixture is solid, durable, non-toxic, and can be used in structural elements. In addition it is a reasonably good insulator and a thermal storage medium. We would not really call this material a hybrid material in a scientific context, more likely it would count to the composite materials. But there are other examples of ancient materials which are closer to the term hybrid material. One impressive case is the old dye Maya blue, which is a mixture of a clay mineral and the organic dye indigo and shows remarkable high stability which indigo alone does not [1,2]. This material is often mentioned when it comes to the beginning of the history of hybrid materials [3].

More modern materials in our everyday life, such as glass or carbon fiber reinforced polymers address our current view on the beneficial combination of two different materials that generate a novel compound showing superior properties compared to the characteristics of the pristine components. Graphically the specific properties of such material combinations can be expressed by diagrams in which different materials features are correlated with each other [4]. These so-called Ashby diagrams give a clear impression how new composite or hybrid materials can fill gaps in material properties that cannot be filled by traditional materials. One example is shown in Figure 1 where the Young's moduli and densities of metals and polymers, and polymer-matrix as well as metal-matrix composites are shown. The composite materials occupy areas of property space that were previously unoccupied by bulk materials. From the diagram it can be concluded how new lightweight mechanical structures can be prepared.

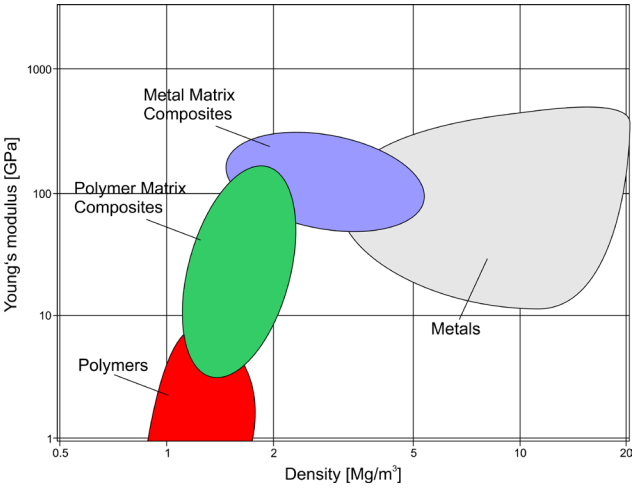

Figure 1: Young's modulus and density of polymers and metals. Combining these materials create polymer and metal matrix composites which fill a previously empty hole in material-property space. Graphics adopted from [4].

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From these considerations it becomes clear that it is beneficial to combine different materials on the macroscopic scale. In materials science sometimes macroscopic composites are already described as hybrid materials [4]. However, chemists would consider such materials as macroscopic composites. The term hybrid material, from a chemist point of view, relates to a material that shows the composite structure on a molecular length scale.

In the past there were many attempts to limit the term hybrid material to specific materials composition or features. One current definition what we understand under a hybrid material is given by the International Union of Pure and Applied Chemistry (IUPAC): *"A hybrid material is composed of an intimate mixture of inorganic components, organic components, or both types of component. Note: The components usually interpenetrate on scales of less than 1 µm"*[5]. This very broad classification has the disadvantage that it still covers a wide range of materials including many types of inorganic crystalline materials, polymer blends, and nanocomposites. A common more specific definition would be: *A hybrid material consists of at least two components – usually an inorganic and an organic component – that are molecularly dispersed in the material.* Following this definition hybrid materials are a special case of composites showing a mixture of the different components on the molecular length scale. In scientific literature there is no clear distinction between the terms hybrid material and nanocomposites. However, for some scientists – including me – there is a clear difference between both types of materials on the basis of structural features [6]. In nanocomposites there is at least one structural element in the nanometer length scale which means in the range of 1-100 nm. On a first view this definition seems not to be far away from the IUPAC explanation of hybrid materials, but the intimidate mixture of the components on the molecular scale can change many properties compared to those of nanocomposites. For example optical and mechanical properties can be very different although similar components are mixed.

In the last decades hybrid materials were developed to such an extent that they belong to the well-established materials nowadays with a high degree of fundamental research going on and also a lot of realized economic applications. The rise in publication numbers and patent applications from the 1980s until the year 2013 is tremendous (Figure 2) and every year new materials, scientific visions, and applications are added.

From the above mentioned points it seems clear that hybrid materials as well as nanocomposites are artificial systems developed in chemistry and materials science laboratories. In fact this is not true because Mother Nature has a clear lead in the structure and function of this type of materials [8,9]. We are currently not able to

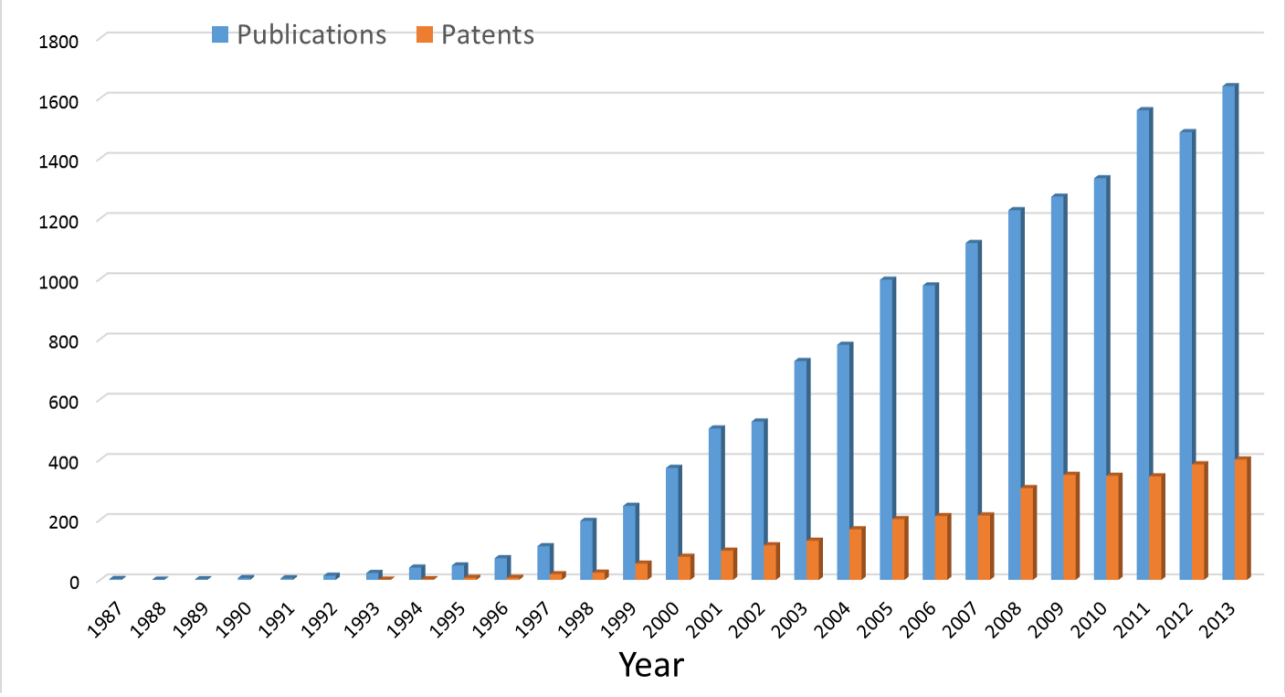

Figure 2: Development of the number of publications and patents in the field of inorganic-organic hybrid materials in the last decades. Source: SciFinder Scholar, Keywords: inorganic organic hybrid materials [7].

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imitate the complex hierarchy of natural hybrid materials and nanocomposites. One of the attempts to copy the perfect development of functional materials in nature is the mimicking of nacre, which is a natural material made from platelets of aragonite glued together by biopolymers[10]. But still we are only in the beginning of the understanding how natural materials are assembled and how we can use these biological concepts.

This short review cannot cover all studies and ideas mentioned in literature within the term hybrid materials. Instead the diversity of the field is shown in short chapters. The interested reader is referred to the cited literature – mainly reviews, to get a fast insight in the field. In addition there are many excellent books, special issues of scientific journals, and reviews available that give a good overview of the scope of the topic [11-22].

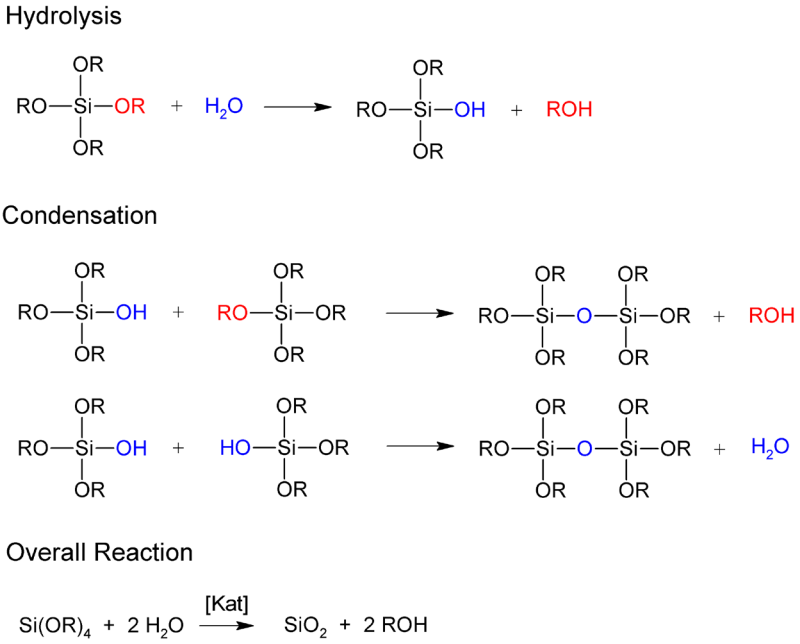
In our view on the development of hybrid materials we will start with the potential origin of the expansion of this topic, which was the sol-gel process. This process and the scientists who were dealing with it in the last 30 years play not only a leading role for hybrid materials but also in nanotechnology. The sol-gel community was from the beginning an interdisciplinary agglomerate of scientists from different branches such as chemists, materials scientists, physicists and even biologists who explored a process from different viewing angles for the benefits of the different disciplines.

2 Methodological concepts in the formation of inorganic-organic hybrid materials

2.1 Sol-gel-based hybrid materials

One of the most widely studied types of inorganic-organic hybrid materials are silicon based sol-gel materials. The developments in sol-gel science and technology particularly in the 1980s can be called the birth of hybrid materials [23]. The sol-gel process starts with a molecular precursor, which forms an inorganic 3D network over time [24-26]. With other words molecular compounds react with each other to build a bulk material connected by strong chemical bonds in the end. Compared to classical solid state reactions the method has the advantage that the precursors react with each other in a liquid environment which reduces many diffusion problems that exist in solid state processes. The latter therefore often require long reaction times and high temperatures.

The basic reactions in the most investigated silicon-based sol-gel process are the hydrolysis of the precursor and the condensation of the formed silanol groups (Scheme 1), which subsequently leads to the formation of a sol and after crosslinking the solid components in the sol to the formation of a gel. This solid product still contains



Scheme 1: Hydrolysis and condensation reactions in the sol-gel process.

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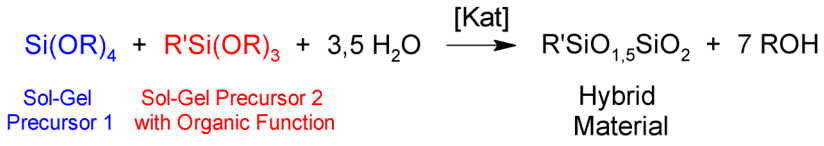
water and alcohol in its pores. Further processing can lead to powders – so called xerogels, thin films, or aerogels [27,28].

In principle the process is a 3D polycondensation reaction which does not lead to a linear polymer but a network structure. In silicon based sol-gel chemistry a catalyst is required to obtain the material in reasonable time. The catalyst – either an acid or a base – influences the morphology of the final material, which means that either a particle-like structure is formed or an extended polymer-like structure. Typical silicon precursors are tetramethoxysilane (TMOS) or tetraethoxysilane (TEOS). The thus produced compounds are pure inorganic materials. However, due to the fact that the process can be carried out at mild temperatures and in usual organic solvents it is possible to entrap organic molecules in the formed inorganic matrix [29]. The formed materials often show much better stability of the organic moieties compared to the free compounds. In addition the inherent porosity of the sol-gel matrix allows the diffusion of molecules to the entrapped compounds, which makes the materials ideal for catalytic or sensor applications [30-33]. Soon it was clear that not only simple organic molecules but also complex biological systems, such as enzymes or proteins can also be entrapped in the inorganic matrix. If the synthetic conditions are chosen correctly the biologic activity can be retained and such systems can be used in biocatalysis or sensing [34,35]. The mentioned materials are based on physical entrapment of organic or biological structures in the porous network without strong chemical interactions – such as chemical bonds - between organic/biological entrapped molecules and the inorganic matrix. But the sol-gel process allows a further strategy. Contrary to Si-O-C bonds Si-C bonds are stable towards hydrolysis, which means that the incorporation of organic groups in a sol-gel network is possible if alkoxysilanes of the type $(RO)_{3-x}SiR_{1+x}$ are applied as co-precursors (Scheme 2).

With this procedure a broad diversity of materials can be formed with various organic functions being attached to the inorganic network. The organic functions of the trialkoxysilanes used as co-precursors change the hydrophobicity of the material, incorporate new functions, or provide groups that can undergo further chemical reactions (Figure 3). The type of functional groups which can be incorporated in the materials is broad and often the molecules are commercially available. If the required trialkoxysilanes are not available through commercial sources they can be prepared by platinum catalyzed hydrosilation reactions between alkenes and trialkoxysilane $(RO)_3Si\text{-}H$. There is only one limit concerning the composition of the functional group: it has to be stable under the conditions of the sol-gel process (aqueous solution, solvent).

The hybrid materials achieved with the combination of alkoxysilanes are manifold and it would be difficult to mention all fields of technological applications. Some examples are the incorporation of chromophores in an inorganic matrix [36-38], the formation of column materials for chromatography [39-41], or the synthesis of coatings with special mechanical properties or permeability [42-45].



Scheme 2: Synthesis of organically functionalized silica materials.

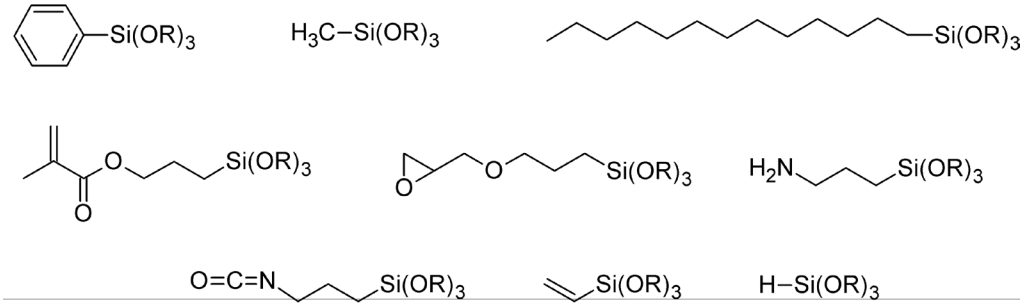


Figure 3: Trialkoxysilanes often used in the sol-gel approach for the preparation of hybrid materials.

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Most of the mentioned hybrid materials are silicon based, however the advantage of the sol-gel process is that different precursors can be mixed in one material. Therefore various metal alkoxides can also be incorporated in the inorganic matrix to provide additional functions, such as mechanical, electronic, or optical properties. One of the problems is that the sol-gel process is a kinetically driven process therefore small differences in reaction kinetics of different precursors can lead to phase-separation or precipitation of one species. Therefore the chemical parameters have to be well-controlled to obtain a homogeneous material [46,47]. There is another problem in the formation of hybrid materials from metal alkoxides: it is more difficult to incorporate organic functions. Contrary to the hydrolytical stable Si-C bonds many metal bonds do not show such stability. Therefore if an organic function has to be incorporated which should be attached to the metal other techniques have to be used. One of the most common methods is the use of coordinating multidentate ligands, such as acetylacetone and its derivatives or organophosphorous compounds [48-51]. Another approach is to use a mixture of metal alkoxides and trialkoxysilanes to form hybrid materials. In this approach the organic function is incorporated by the trialkoxysilane. Metals are integrated in a hybrid material because of their specific properties, for example the refractive index can be increased [52,53], thus new materials for optical waveguides can be produced [54,55], or materials for photovoltaic applications [56]. An additional difference to a pure silicon-based hybrid sol-gel material is that metal oxides often form crystalline structures even under the ambient conditions of the sol-gel process while the silica materials are generally amorphous.

2.2 Polymer based hybrids

The organic components in inorganic-organic hybrid materials are either small organic molecules or macromolecular compounds. These polymer-based hybrid materials can be formed by different approaches. Either the sol-gel process is applied in presence of a polymer with or without chemical bonding between the two components, or inorganic components are incorporated into the polymer as molecular building blocks [57,58]. Most of the times chemical interactions between the organic polymer and the inorganic component are preferred, because this usually leads to more stable and highly homogeneous materials. Various strategies can be used to form such polymeric systems; one of the most applied approaches is the in situ polymerization of the organic polymer in presence of the inorganic moiety. The broad diversity of polymers that can be used in this approach in addition to the well-established methods for polymer processing makes this material class very interesting for many technological applications. To get homogeneous systems it is important that the polymer and the inorganic components show a high compatibility which is usually achieved by some kind of chemical interaction. This can either be van-der-Waals forces, hydrogen bridges, coordinative or covalent bonds. Hence either the polymer chain provides enough compatibility to the often hydrophilic inorganic species or the inorganic components are adapted to the polymer chain.

2.3 From compact materials to tailored porosity

As already mentioned above the sol-gel process works at quite mild conditions and thus the incorporation of additional molecules during the reaction is possible. One of the major approaches in structuring of sol-gel materials is the cooperative self-assembly of surfactant molecules as templates, which is used for the controlled formation of porous materials [59,60]. The surfactants self-assemble in presence of the sol-gel precursors, which form their networks around these self-assembled structures. Generally amphiphilic surfactants or block-copolymers are used in this procedure, which are removed after the network has formed. The resulting mesoporous materials reveal a long range ordering of pores with diameters between 2-10 nm and structural features of the pores that depend on the phases formed by the templates [61,62]. Based on the very regular nature of the pores such materials are interesting in many applications, for example catalysis, separation technology, or drug delivery [63-67]. When the materials were discovered the porous network consisted only of a purely inorganic silica network. The organic functionalization of mesoporous materials made it possible to extend their potential applications.

Three different methods can be used to prepare hybrid mesoporous materials: (i) the postsynthetic grafting of silane coupling agents to the surface of purely inorganic porous materials, (ii) the cocondensation of trialkoxysilanes during mesoporous network formation, or (iii) the use of multifunctional alkoxysilane precursors to form so-called periodic mesoporous organoocsilicas (PMOs) (Figure 4) [68].

The first method is the postsynthetic functionalization of a mesoporous material. Attachment of organosilane

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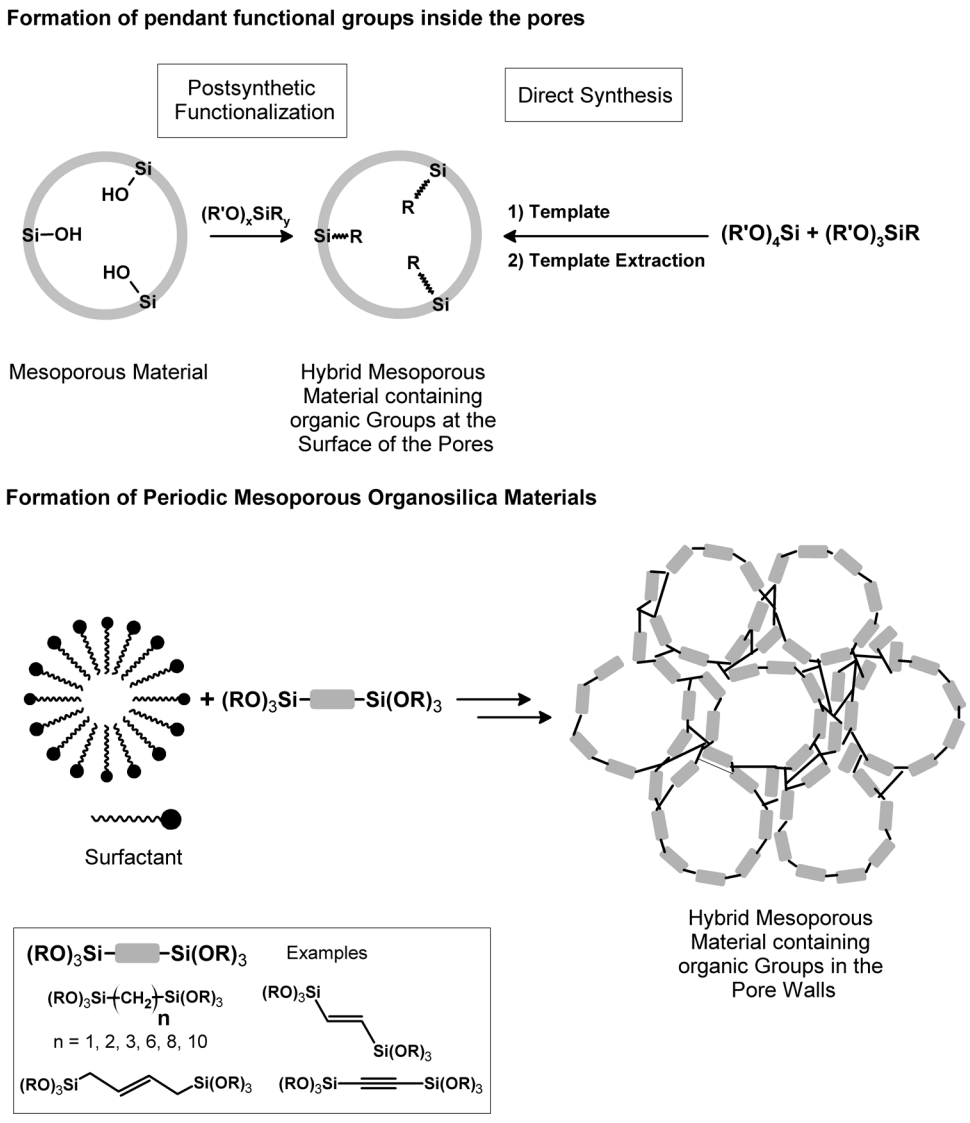

Figure 4: Synthesis of mesoporous hybrid materials applying three different routes. Adopted from [68].

coupling agents of the type $RSi(OR')_3$, $RSiCl_3$,or R_3SiCl to the surface of the pores results in functional mesoporous inorganic–organic hybrid materials [69,70]. This postsynthetic grafting has been widely employed to anchor various organic groups onto the surface of the pores, including organometallic species, amino- and thiol-groups, or reactive sites for polymerizations such as epoxides or methacrylates. However, this method often leads to quite low functionalization rates and inhomogeneous distribution of the functional groups. In addition, the pore volume often decreases dramatically.

The second method to prepare a hybrid mesoporous material is the cocondensation of trialkoxysilanes during the network formation [71-73]. This method leads to higher degrees of functionalization compared to the postsynthetic grafting. A variety of functional groups can be incorporated by this method including phenyl, vinyl, sulfonyl, or amino groups.

The third method to prepare mesoporous hybrid materials is the production of materials that have the organic functions incorporated as part of the inorganic network. This procedure uses bifunctional organosiloxane precursors, also referred to as bridged silsesquioxanes, of the general formula $[(R'O)_3Si]_mR(m \geq 2)$ as the network-forming species. The resulting materials, called periodic mesoporous organosilicas (PMOs), contain the organic groups as an integral part of the inorganic-oxide framework [74-76]. This route provides porous materials with accessible and well-dispersed chemical functions, which in many cases are more stable than those of simply anchored molecules at the surface of the traditional mesoporous materials.

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2.4 Crystalline porous networks from coordination compounds

In recent years a new type of porous materials received a lot of scientific attention which also can be considered as a hybrid material: metal organic frameworks (MOFs) [77]. These crystalline and highly porous solid materials are often prepared by the combination of rigid organic ligands with at least two coordination sites with multinuclear metal building units (Figure 5). The latter act as joints in the 3D network. Therefore these compounds belong to the class of 3D coordination polymers. A rational design of these materials is possible because the size and the chemical environment of the formed void spaces are defined by the length and functionalities of the organic bridges. Due to the wide variability of pore sizes and the very high specific surface area, up to several thousand m^2/g, these materials have attracted much interest as hosts for other molecules. Well-known materials in this class are those containing tetranuclear $Zn_4(\mu_4\text{-}O)$ clusters acting as octahedral joints which are linked by rigid aromatic dicarboxylate ligands to form a primitive cubic structure [78,79]. The most promising potential technological application is their use as gas storage materials [80-86].

2.5 Hybrid materials based on preformed molecular building blocks

Up to now only systems were listed that start with molecular precursors that lose their original molecular identity during the formation of the hybrid materials. For example alkoxysilanes are transformed into silica or silsesquioxanes. Alternatively preformed molecular building blocks can be used that have a chemically stable, relatively stiff structure, which will not be lost during the preparation of the bulk material. In case of silicon-based structures these systems can be prepared for example in a first step and then mixed as the inorganic component with an organic compound to result in a hybrid material.

A prominent example are polyhedral oxo-cluster compounds which consist of a compact body and some reactive sites on the surface that can be used to either compatibilize them in an organic matrix or to connect them to an organic material, such as a polymer, by strong chemical bonds. One class of such precursors is based on silicon and use the high stability of the silicon oxygen bond for the formation of the rigid backbone. These silicon-based precursors can be divided into spherosilicates and silsesquioxanes [6,88]. Both types of precursors are distinguished by their composition. Spherosilicates show the chemical composition of $(SiO_2)_n^{n-}$. The most important example of this precursor class is the system with n = 8. Each corner of these cubes consists of silicon atoms with a tetrahedral oxygen environment (Figure 6). Contrary silsesquioxanes show the composition $(RSiO_{1.5})_n$ which means that each silicon atom has three neighboring oxygen atoms and one organic rest R. Compounds cosisting of very regular three dimensional objects are called polyhedral oligomeric silsesquioxanes (POSS) and have shown much attraction in materials chemistry in the last two decades [89,90]. While the anionic spherosilicates need additional reactions for being transferred to building

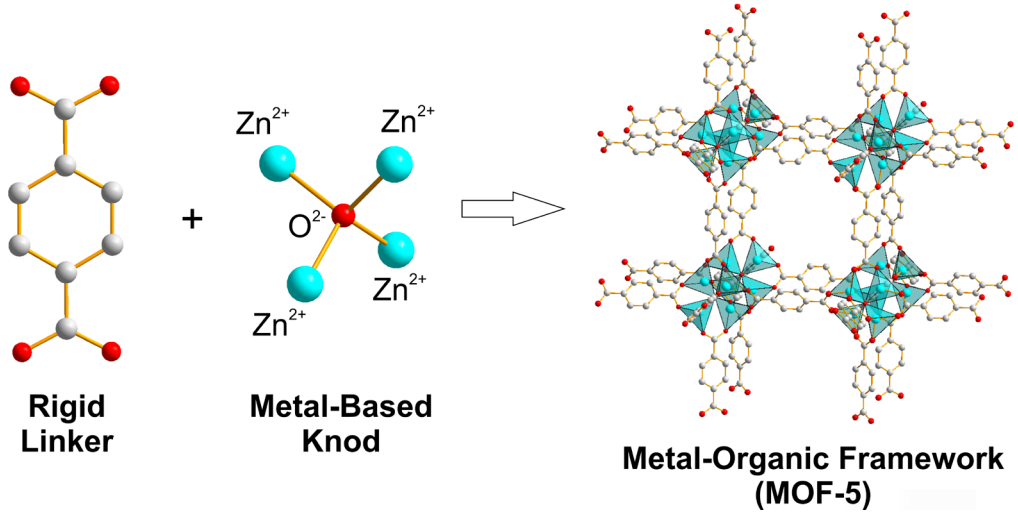

Figure 5: The combination of rigid organic linkers and metal-based knods leads to the formation of metal organic frameworks – a special kind of crystalline hybrid materials. Adopted from [87].

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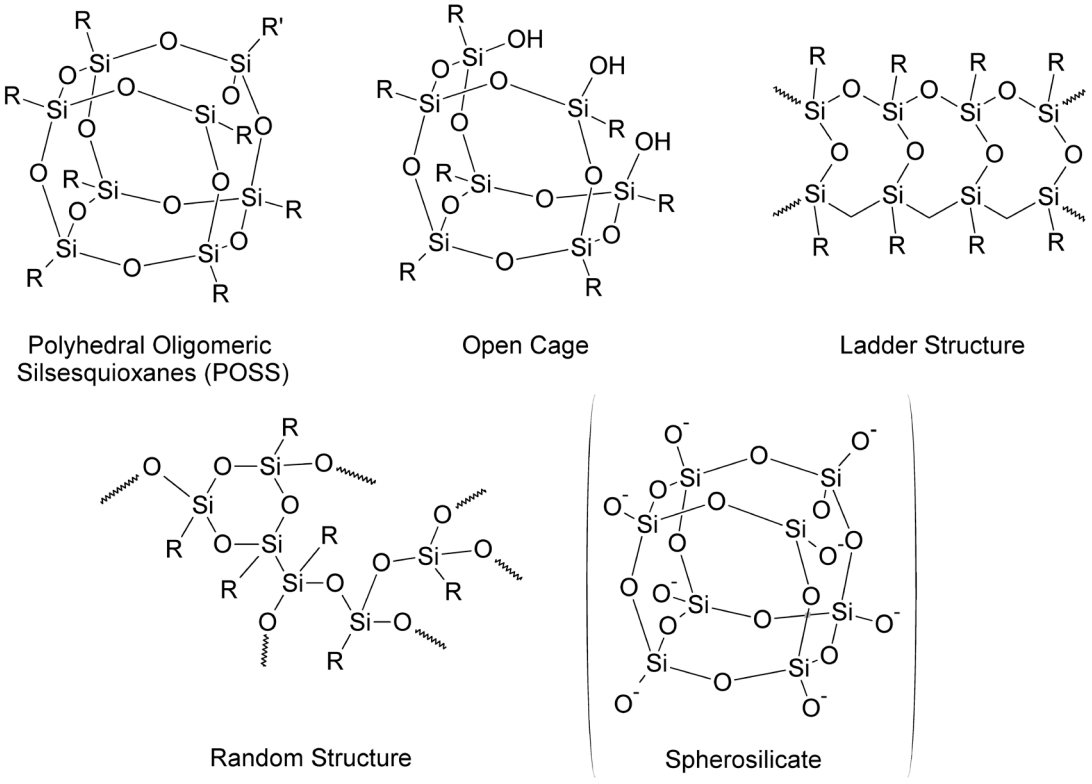

Figure 6: Silicon-based structures which can be applied as inorganic components in hybrid materials.

blocks that are active in organic reactions the R groups in the silsesquioxanes can be directly used for various reactions. Both structures can act as the inorganic components in a hybrid material.

Depending on the kind of substitution pattern POSS systems can be used as comonomers or as crosslinking agents in polymerization reactions. In many cases POSS compounds that are used as monomers consist of one polymerizable organic rest R' and the other groups are used to tailor the solubility in the comonomer. With these precursors usually chain polymerizations can be carried out and the final macromolecular structure is represented by an organic polymer chain with pendant inorganic groups. In the case of crosslinking agents more than one reactive group is available and usually all eight groups can be used for various reactions. With this approach a plethora of materials was already synthesized consisting of well-defined inorganic components showing extraordinary mechanical, optical, and thermal properties [91-94]. The size of these building blocks is around 1 nm in diameter. Therefore they are located at the borderline to nanocomposites.

Besides these polyhedral silsesquioxanes also ladder type polymer-like silsesquioxanes were used in hybrid materials [95,96]. Compared to the cubic building blocks they do not show such high tendencies to agglomerate in polymeric matrices. They behave more like polymers but due to their very stable double stranded chain structure they can be used to improve thermal properties of hybrid materials.

In addition to those silicon based structures also organic functionalized metal oxo-clusters are a prominent example of inorganic molecular building blocks which are not structurally altered during the formation of hybrid materials [97,98]. The systems are usually prepared by simple chemical reactions and surface functionalized in one step. Contrary to the silicon based building blocks, which usually only improve the mechanical and thermal properties of the organic matrix, the metal oxo-clusters can induce novel properties such as a change in the refractive index, magnetic, or electronic properties in the organic matrix.

Beside the mentioned materials there are many other hybrid systems and every day there are new systems developed. This article only shows a small excerpt of the diversity of components which can be applied for the preparation of hybrid materials. In the following chapter there is an overview of potential applications of such hybrid materials.

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3 Technology driven exploration of hybrid materials

3.1 Hybrid coatings

One of the most important advantages of inorganic-organic hybrid materials is their simple processing technology and their modular construction, which allows a tailored design of this materials class for various applications. In addition their processing and composition allows for the production of transparent films, which are ideal prerequisites to use them as coatings [43]. Particularly their broad use on different substrate (ceramics, metals, polymers, etc.) and their low processing temperatures (usually <150°C) makes them ideal for modern coating requirements for example in electronic industry. The first systems used were based on the sol–gel process, by the combination of tetraalkoxysilanes, trialkoxysilanes and metal alkoxide precursors (Figure 7). Particularly the Fraunhofer-Institute of Silicate Chemistry in Würzburg (Germany) and the Leibniz Institute of New Materials in Saarbruecken (Germany) contributed strongly to the coating technology exploration of the sol-gel process. The different components in the coatings fulfill specific duties, organic groups can be used for a better wetting and adhesion of the coating to the substrate, or they can be additionally crosslinked by radiation with UV light [99]. The inorganic moieties like Si or metal alkoxides increase the inorganic crosslinking and thus the stiffness of the material, induce higher refractive index, or change the porosity of the coatings. By fine-tuning the composition of the coatings, the properties can be changed which allows the adaptation of basic compositions towards special applications, like scratch resistant coatings. Some applications of such sol-gel coatings are in the field of abrasion resistant systems on polymers [100-102], the induction of antistatic properties on substrates [103-105], antireflective [106-108], and high barrier coatings for oxygen and water [42,109,110].

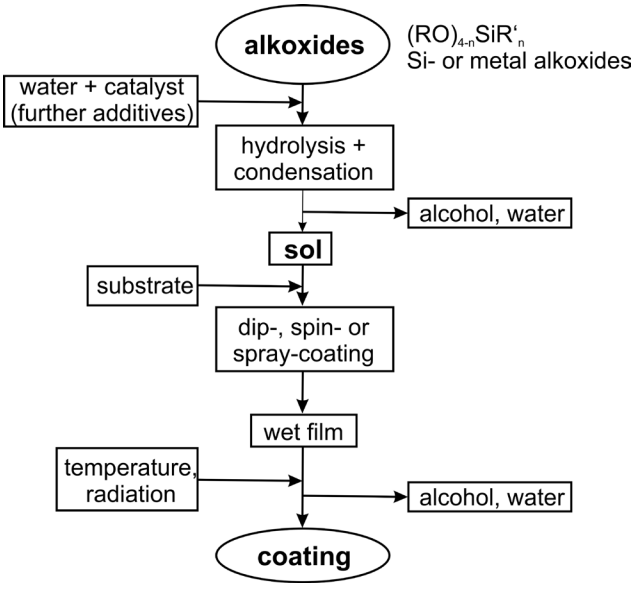

Figure 7: Sol-gel processing for coatings. Adopted from [43].

New corrosion resistant coatings were one major target of applied research in the last years. This is due to the recent revival of interest in industrial applications of metals and alloys based on magnesium and aluminum. Particularly lightweight structural applications in the automotive, aerospace, and electronics industries are the field of application of compounds. Inorganic-organic hybrid systems are one of the major strategies to form coatings on these materials. The sol-gel coating method allows the production of passive as well as active corrosion protecting coatings which also show excellent abrasion resistance [111-115]. Therefore this method offers many advantages compared to conventional technologies.

3.2 Hybrids for optical applications

Based on the high transparency which can be achieved for inorganic-organic hybrid materials and the high mechanical flexibility compared to traditional glass, there is a large interest in novel materials for optoelectronic devices. Although a hybrid material belongs to the materials class of composites, which usually do not show a high degree of transparency because the different components included scatter light due to the size of the components and variations in refractive index, it is astonishing that inorganic-organic hybrid materials virtually do not show a scattering of light. One of the reasons is the molecular dispersion of the different components in the material. The structural units are much smaller than the wavelength of light and thus the scattering is minimized. In addition modern materials for optoelectronic devices require higher thermal stability and a better compatibility to processing techniques used in conventional semiconductor devices production, for example photolithographic processes. Inorganic–organic hybrid polymers seem to be ideal for such applications. The materials can be functionalized such that their physical and chemical properties can be tailored towards, e.g. optical applications on wafer-scale such as waveguides, gratings or micro optical devices [116]. Examples of optical devices produced with inorganic-organic hybrids are high-refractive-index materials [52,117], photochromic

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materials [118], waveguide materials [55,119], nonlinear optical materials [120], and photodetectors [121].

High refractive index thin films were, for example, obtained by the incorporation of metal oxide structures like titania or zirconia [52]. The refractive index, which is around 1,5 for usual organic polymers and silica can be increased to values above 2 by applying such approaches. A large future market will be optoelectronic packaging of LEDs and photovoltaic devices where inorganic-organic materials also already showed a great potential [122].

3.3 Hybrids in electronic applications

There is an increasing need for new materials in electronics industry based on the continuous need to increase integrated circuit performance through shrinkage of the circuit elements. Traditional materials in the electronic industry, such as lithographic tools or photoresist materials have reached their limits. Therefore, continuing scaling will require the introduction of new materials. Inorganic-organic hybrid materials can offer a solution. They are particularly suited for electronic applications because their precursors can be purified to a very high degree based on chemical approaches and therefore impurities influencing the electronic performance can be minimized. In addition their processing is highly reproducible and partially also lithographic tools can be used for their patterning. Examples of the use of hybrids in electronic devices are low-k dielectrics [123,124]. In these materials often silicon species play a major role and there has been some attempt to use POSS derivatives for the production of electronic materials [93].

Besides this small overview of technological applications of hybrid materials there is a large variety of other promising applications. Due to the modular composition of the hybrid materials a plethora of components seem to be suitable for mixing. Therefore there seem to be no limits in the vision of future applications.

4 Conclusions

The field of hybrid materials is one of the most exciting research areas in science and technology in the last three decades. Many different routes to change the composition and to control the structure of this materials class were developed in fundamental research and transferred to technological applications. Particularly the combination of functional inorganic with functional organic and biological components made hybrid materials to probably the broadest playground of different disciplines in materials science. Promising properties of the materials, such as high optical transparency, improved stability compared to their pristine organic components, and the availability of different functions in one material, make hybrid materials the most promising material classes in various emerging technologies such as optoelectronics, energy production and storage, and lightweight materials for transportation technologies.

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