Molecular Imprinting Science and Technology: A Survey of the Literature for the Years from 2004 to 2011

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Summary

Herein we present a survey of the literature covering the development of molecular imprinting science and technology over the years 2004 to 2011. In total, 3779 references to the original papers, reviews, edited volumes and monographs from this period are included, along with recently identified uncited materials from prior to 2004 which were omitted in the first instalment of this series covering the years 1930 to 2003.[1] In the presentation of the assembled references, a section presenting reviews and monographs covering the area is followed by sections describing fundamental aspects of molecular imprinting including the development of novel polymer formats. Thereafter, literature describing efforts to apply these polymeric materials to a range of application areas is presented. Current trends and areas of rapid development are discussed.

Keywords: Molecular imprinting, molecularly imprinted polymer, template polymerization

Abbreviations used:

CE: capillary electrophoresis

CEC: capillary electrochromatography

LC: liquid chromatography

MIP: molecularly imprinted polymer

MISPE: molecular-imprint based solid-phase extraction

NMR: nuclear magnetic resonance spectroscopy

SPE: solid phase extraction

INTRODUCTION

The continued increase in the volume of literature describing the design, development and application of molecularly imprinted polymers (MIPs) reflects the maturation of this field of study and the broad interest it has attracted from the scientific community in general, Fig. 1.

Insert Figure 1 about here

Figure 1. The number of publications within the field of molecular imprinting science and technology per year for the period 1931–2011.[2]

We previously[1] presented a comprehensive survey of the field spanning the literature from the seminal work of Polyakov in the 1930s,[3] until 2003. In this, the second instalment in this series, we cover first reviews, monographs and contributions to edited volumes; followed by work dealing with fundamental aspects of molecular imprinting; the development of novel polymer formats and attempts to apply these polymeric materials to a range of application areas. We conclude with a brief commentary regarding prospects for future developments within this exciting and

dynamic field of research endeavour. Papers are only cited once, and our classification of papers is based upon our joint assessment of the perceived primary area of impact of each paper.

We have included all material known to us to have been published within the timeframe covered by this survey. With the exception of a limited number of papers of historical significance, we have limited the survey primarily to works published in English. It should be stated that the past few years have witnessed a dramatic increase in the volume of molecular imprinting related literature published in Chinese, and the appearance of some English translations of some key Chinese journals. Furthermore, a number of reports from the period prior to 2004 that were not included in the previous survey have been identified and are herein presented. Of particular historical interest is a series of eight publications in German from the late 1960s by Rackow and colleagues describing efforts to develop non-covalent molecularly imprinted materials using condensation polymers.[4-11]

A small number of errata, corrigenda, comments and replies to comments have been published in the period covered by this review. These are cited immediately following the original paper, with a single exception,[12] which relates to a paper cited in our previous survey[1] as reference number 34.

For the purpose of this survey we have elected to define molecular imprinting as:

"*The construction of ligand selective recognition sites in synthetic polymers where a template (atom, ion, molecule, complex or a molecular, ionic or macromolecular assembly, including micro-organisms) is employed in order to facilitate recognition site formation during the covalent assembly of the bulk phase by a polymerization or polycondensation process, with subsequent removal of some or all of the template being necessary for recognition to occur in the spaces vacated by the templating species*".[1]

Schematically this can be represented by Figure 2.

Insert Figure 2 about here

Figure 2. Highly schematic representation of the molecular imprinting process:[1] The formation of reversible interactions between the template and polymerizable functionality may involve one or more of the following interactions: [(A) reversible

covalent bond(s), (B) covalently attached polymerizable binding groups that are activated for non-covalent interaction by template cleavage, (C) electrostatic interactions, (D) hydrophobic or van der Waals interactions or (E) co-ordination with a metal centre; each formed with complementary functional groups or structural elements of the template, (a-e) respectively]. A subsequent polymerization in the presence of crosslinker(s), a cross-linking reaction or other process, results in the formation of an insoluble matrix (which itself can contribute to recognition through steric, van der Waals and even electrostatic interactions) in which the template sites reside. Template is then removed from the polymer through disruption of polymer—template interactions, and extraction from the matrix. The template, or analogues thereof, may then be selectively rebound by the polymer in the sites vacated by template, the 'imprints'. While the representation here is specific to vinyl polymerization, the same basic scheme can equally be applied to sol-gel, polycondensation etc.

REVIEWS AND MONOGRAPHS

The number of reviews on the topic of molecular imprinting has continued to increase[1,13-214] which is a direct reflection of the rapidly expanding primary literature covering the area. The increasing incidence of molecular imprinting related material in reviews from other fields indicates that molecular imprinting science and technology has established itself within the broader scientific community.[215-501] Similar trends are observed in the cases of multi-authored monographs and general scientific reference works.[502-627] Since the initial survey, a number of books dedicated to the topic have been forthcoming, these provide the scientific public with consolidated presentations of various aspects of the design, preparation, characterization and application of molecularly imprinted polymers.[628-681]

FUNDAMENTAL ASPECTS

While the evolution of the field runs apace, there is undoubtedly a significant and continuing need for improved fundamental understanding of the mechanisms underlying the molecular imprinting effect and their consequences for the nano-, micro- and macro-level structure and function of imprinted materials. The establishment of a capacity to predict material structure and function is possibly the single factor most likely to hasten the development of molecular imprinting. Over recent years, a range of theoretical and experimental strategies has been deployed to address fundamental aspects of MIPs at nano-, micro- and macro-levels. These efforts, summarized below, have yielded new insights, novel tools, and new formats,

the latter, in particular, having had considerable impact on the development of MIP application areas.

Theoretical and Computational Studies

Recent years have seen significant progress in the development and application of theoretical and computational strategies for the elucidation of the mechanisms underlying MIP synthesis and ligand-polymer recognition events, and as a tool for MIP design.[682-768] The impact of these techniques is seen in the increasing frequency with which they are being used as a predictive tool in the development of new MIP systems. We suggest this trend shall continue and that the further development of computational methods and increases in computing power shall help drive this process.

Spectroscopic Studies

Traditionally, spectroscopic methods have been invaluable for the study of fundamental aspects of the molecular imprinting process, in particular events taking place in prepolymerization mixtures. The work of recent years[769-805] has seen a continued use of NMR, IR and UV-Vis spectroscopies for characterizing monomer-template interactions, to the point where such studies are becoming essential aspects of MIP design, either for the screening of monomers for interaction with template, or for the validation of computational design data. A recent trend is the significant increase in the number of spectroscopic studies of ligand-MIP interactions. To these ends, Raman spectroscopy, light scattering, circular dichroism and various fluorescence and chemiluminescence based techniques have also been deployed. The use of XAFS, diffraction studies, XPS and high resolution AFM has become more prominent. AFM and various fluorescence techniques have played important roles in the characterization of thin-film MIPs. We perceive that these trends shall continue, especially for probing polymer structure and for the direct study of polymer-ligand binding events.

Recognition

The demonstration of template (or template analog)-selective recognition by a MIP remains a cornerstone in the development of all new MIP systems. Various strategies have been employed to characterize ligand-polymer binding events,[806-1196] which have in turn provided fundamental insights, and in some cases inspiration, for the development of new applications; such as protein-imprinted hydrogels capable of nucleating protein crystallization.[1197,1198] Radioligand binding studies and chromatographic, electrochemical, gravimetric and spectroscopic based analyses, or combinations thereof, have been important for demonstrating molecular imprinting effects.

Formats

The development of alternatives to the synthesis of traditional monolithic polymers has been driven by both practical considerations, for example: ease of scale-up, requirements for integration with other technologies, such as sensors, and for performance issues, e.g. mass transfer or flow properties. Methods for preparation of discrete MIP particles, composites and molecularly imprinted polymer surfaces and fibers are areas that have been gaining in prominence over recent years and now constitute a significant portion of the original literature published in the molecular imprinting field.[1199-1704] The importance of new methods for MIP synthesis and regulation of polymer morphology and structure in general, is a critical factor in the development of materials for new applications.

APPLICATIONS

Historically, the development of molecularly imprinted polymer science and technology has been driven by application development, as reflected in the abundant patent literature covering the area. The dramatic increase in the volume of scientific literature in the field has been closely paralleled by developments in the patent literature. Interestingly, while companies and governmental agencies are well represented in the MIP intellectual property sector, it is academic institutions that have so-far been the greatest contributors. Here we categorized application oriented papers under seven application headings.

Solid Phase Extraction

The development of MIPs as selective adsorbents in solid phase extraction (often referred to as MISPE) was a natural progression from early work aimed at developing MIP chromatographic stationary phases. This is the application area that has found most commercial viability, as witnessed by the presence of several products on the market. The bulk of the MISPE studies reported to date[1705-2331] has been directed to (small) organic structures, with substances of clinical and environmental impact of particular interest. SPE systems for analysis of ionic and inorganic materials have also received considerable attention.[2332-2535] As the demand for more sensitive analytical techniques continues to grow, so too should the need for robust technologies such as MISPE. Access to MIPs in new formats should further improve the use of these selective materials.

Assays

Efforts to employ MIPs in assays has extended well beyond the large volume of literature focused upon MIPSE, most notably capillary electrophoresis (CE) and capillary electrochromatography (CEC),[2536-2588] and liquid chromatography (LC).[2589-2722] In contrast to the 90's and early 00's, the proportion of studies devoted to, or heavily dependent upon, liquid chromatography (LC) has reduced significantly. In the cases of CE and CEC, however, interest in these techniques has been sustained. We suggest that this development is in part fuelled by the sensitivities of these techniques and in part by developments in the range of chemistries used for preparing MIP-containing capillaries.

A number of papers report assays based upon competitive ligand binding studies.[2723-2763] The high sensitivities of assays using either radiolabeled or non-isotopically labeled studies (fluorescence and chemiluminescence) afford access to events taking place at very low concentration regimes that are not generally accessible with chromatographic methods. Interestingly these methods appear to be being superseded by the development of chemical sensors and biosensors based on MIPs where the binding event gives rise to a measurable response by various transduction mechanisms.

Sensors

The application of MIPs in the development of sensors has continued to flourish. It could be argued that the increased use of MIPs in sensor development has been in part driven by developments in MIP formats, not least by the establishment of reproducible strategies for thin film, fiber and (nano)particle preparation. Optical,[2764-2995] electrochemical[2996-3310] and acoustic[3311-3430] sensor platforms are those most frequently reported in conjunction with MIP recognition elements are based upon either platforms. The potential of molecular imprinting in sensor development is reflected in the current rapid growth in the number of reports of devices with MIP-based electrochemical transduction mechanisms. The appearance of a limited number of studies using calorimetric[3431-3434] and mass spectrometric detection[3435,3436] over recent years may provide an indication of areas were MIP-based sensors may find increasing use in the future.

Membranes

The majority of reported molecularly imprinted membrane studies have as an ultimate goal either process development (in particular for separation and purification) or incorporation with sensors. [3437-3554] While efforts to develop and apply molecularly imprinted membranes continue, it remains clear that more work is required to address the mechanisms underlying membrane function.

Catalysis and Synthesis

While much has been achieved in terms of the development of MIPs capable of recognizing a given molecular species, relatively few examples exist of polymers with enzyme-like activity. In principle, a MIP with enzyme-like activity should recognize substrate(s), orchestrate the making and/or breaking of bonds, and expel the product to prepare for a new cycle. This may rightly be deemed a daunting task. Nonetheless, MIP-based catalytic systems and the use of MIPs in synthesis continues to evolve.[3555-3672] Recent reports of several systems with significant catalytic power bodes well for the continued development in this sector.

MIPs meet biology

The integration of MIPs in, or interaction of MIPs with, biological systems is perhaps the area that has drawn most attention from the scientific community at large over recent years. Two themes dominate this sector, namely the use of MIPs for drug delivery[3673-3737] and the development of MIPs with biological function.[3738-3777] The use of molecular imprinted materials in conjunction with ocular drug delivery is arguably the sector that has come closest to clinical use. Perhaps even more challenging is the concept of developing MIPs that interact directly with biological systems; efforts to this end appear to be gaining in pace and number. The first use of MIPs in vivo,[3778] for sequestration of a toxin, constitutes a highly significant breakthrough for the field as a whole, Fig. 3. Finally, the idea of molecular imprinting-related processes constituting a possible mechanism for the creation of Life has been proposed.[3779]

Insert Figure 3 about here

Figure 3. The sequestration of the toxic peptide melittin (a component of bee venom) from the blood stream of living mice by MIP nanoparticles (NPs) presented by Hoshino and Shea (178, 3778). a) Survival rates over a period of 24h following the intravenous administration of melittin (4.5 mg/Kg). NPs (30 mg/Kg) were administered 20s after the toxin: MIPs (red), NIPs (grey), control group (green). Significant survival rates were seen in the case of MIP-adminstered mice (Wilcoxon P values). b) Fluorescent imaging of Cy5-labelled melittin shows that binding to MIP NPs results in clearance of the toxin from the peripheral circulation and transportation to the liver in the treated mice (right) while in the untreated control group (left) the toxin remains widely distributed throughout the body. c) Fluorescence intensity imaging of the livers of mice treated with different concentrations of Cy5-melittin and MIP NPs. Reproduced from Ref. 189 with permission from The Royal Society of Chemistry.

SUMMARY AND FUTURE OUTLOOK

The continued growth of research activity within the field of molecular imprinting is reflected in the dramatic increase in the scientific literature covering the area over recent years. While significant improvements in our understanding of the molecular imprinting process have been achieved, and many new types of polymer, polymer format and application area have been investigated, a number of significant fundamental challenges regarding the mechanisms underlying the technique remain to be explored and many opportunities for applications are still to be exploited. Accordingly, we envisage a continued growth in research activity in the area over the coming years, and a concomitant increase in the volume of literature dealing with the science and technology of molecular imprinting.

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