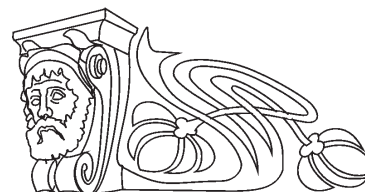




Review

## Molecularly imprinted polyaniline: Synthesis, properties, application. A review



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**Abstract.** Molecular imprinting is a rapidly developing and promising approach for the selective recognition for target molecules of different nature. In this review, we have collected works devoted to synthesis and application of polyaniline-based molecularly imprinted polymers (MI-PANI) over the last 5 years. The manuscript provides brief descriptions of the main approaches to the synthesis of PANI MIPs and the advantages and disadvantages of each technique. We also discuss the effect of various factors on the process of MI-PANI synthesis, including polymerization methods, molecular weight of template molecules and the types of scaffolds. The analytical characteristics of the resulting sensors are also provided. Thus, it can be concluded that polyaniline is a very promising material for MIPs synthesis.

**Keywords:** polyaniline; molecular imprinting; molecularly imprinted polymers; sensors

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Обзорная статья

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**Полианилин в молекулярном импринтинге: синтез, свойства, применение. Обзор**

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**Аннотация.** Молекулярный импринтинг является быстро развивающимся и перспективным подходом селективного распознавания молекул-мишеней различной природы. В обзоре собраны работы, посвященные синтезу и применению молекулярно-импринтированных полимеров на основе полианилина (МИ-ПАНИ) за последние 5 лет. Приведено краткое описание основных подходов к синтезу МИ-ПАНИ, а также рассмотрены их преимущества и недостатки. Обсуждено влияние различных факторов на процесс синтеза МИ-ПАНИ, в



том числе метода полимеризации, молекулярной массы молекул темплата и типа подложки. Особое внимание уделено аналитическим характеристикам сенсоров на основе МИ-ПАНИ. Показано, что полианилин является перспективным материалом для синтеза МИП.

**Ключевые слова:** полианилин; молекулярный импринтинг; молекулярно-импринтированные полимеры, сенсоры

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## Introduction

In recent years, molecular imprinting technique has become a powerful tool for the synthesis of lock and key systems that are very effective at capturing the target molecules. Molecularly imprinted polymers (MIPs) are highly cross-linked polymers containing artificially created cavities called recognition sites that selectively bind template molecules [1, 2, 3]. As synthetic receptors, MIPs are characterized by high stability, durability, and low synthesis costs. More than 10 000 molecules with different structure have been used as template molecules for the MIP synthesis [2], including inorganic ions [4], drugs [5], pesticides [6], proteins [7, 8], viruses [9], cellular structures [10] and various macromolecules and microorganisms [11, 12]. The current theoretical and experimental knowledge offers an opportunity to develop MIP-based systems to address a wide range of challenges in science and technology including analytical chemistry and separation science [13].

MIPs are based on polymers of various nature. There are polymers of natural origin, such as chitosan obtained from chitin [14], but the most common polymers used for MIP synthesis are those that contain monomers with basic (vinylpyridine), acidic (methacrylic acid) and hydrogen bonds (methacrylamide) or hydrophobic groups (styrene). Usually, MIPs are electrically insulating polymers, that may lead to low sensitivity and selectivity of analytical systems due to the electron transport barrier. This disadvantage of MIPs can be overcome by using conductive polymers with characteristic electrochemical activity [15]. Their synthesis can be carried out using simple, versatile and cost-effective approaches. Conductive polymers can assemble into supramolecular structures [16] turning them into a prospective matrix for MIPs design. One of the most interesting conductive polymers is polyaniline (PANI), characterized by good biocompatibility, high physical and chemical stability, adaptability of synthesis and high electrical conductivity. PANI is capable of self-assembly, leading to increases in the surface area to volume ratio, a fact that is relevant to the case of MIPs development. Moreover, PANI

is the only conductive polymer whose electronic structure, magnetic and optical properties, electrical conductivity and structural features can be adjusted by doping–dedoping process [17].

In this review, we considered the studies of the molecularly imprinted polyaniline (MI-PANI) over the past 5 years. We compared various methods of MI-PANI synthesis, and discussed their prospects and application fields.

## Synthesis of Molecularly Imprinted Polyaniline

The synthesis of MIPs is based on the formation of a complex between the template molecule and the functional monomer through covalent or non-covalent interactions, followed by the removal of the template molecules from the polymer network. The functional monomer determines the method of polymerization, including electropolymerization, chemical polymerization, creation of composite membranes (MIPs particles and conductive material), phase inversion, lithography and surface stamping [1, 18].

PANI is a widely studied and the most interesting example among conductive polymers for MIPs synthesis. There is a large number of studies describing the properties of PANI, its synthesis methods and applications [19, 20]. The morphology of PANI structures ranges from nanoparticles (NPs) to micron-sized clusters. Depending on the purpose of the further application, PANI can be synthesized in different forms, such as films [21, 22, 23], nanotubes [24], nanospheres [25], nanowires [26], nanofibres [27, 28, 29] and multicomponent structures [30]. Much experience has been gained over the recent decade regarding the synthesis of different PANI structures that can be adapted to MIPs creation.

The important features of PANI are simplicity and low cost of synthesis compared to other polymers and commercial availability of reagents. Other benefits of PANI include low toxicity, high conductivity, good redox reversibility and environmental resistance [19, 31]. These properties provide the opportunity to develop low-cost analytical systems based on MI-PANI that meet the requirements of “green chemistry”.



There are two main methods of MI-PANI synthesis [19]: electrochemical and chemical polymerization of aniline in the presence of various oxidants. The choice of the method depends upon the task and

various forms of MI-PANI can be obtained by varying the polymerization conditions. Comparison of electrochemical and chemical oxidative polymerization methods is provided in Table 1.

Table 1

**Electrochemical and chemical polymerization of aniline**

Comparison	Electrochemical polymerization	Chemical polymerization
Advantages	High purity of the product High product yield (wt. %) Synthesis process control	Simplicity of synthesis Wide choice of oxidants Wide scaffold types Semi-industrial yield of product
Disadvantages	Limited electrode surface area Requires electrically conductive scaffold Leaching of MIPs	The complexity of synthesis monitoring Costly and time-consuming purification of the product
Morphology of MI-PANI	Nanowires [32, 33] Film [21, 31, 34, 35] Nanofibers [28, 29]	Nanoparticles [33, 34, 35, 36, 37, 38] Film [22, 23, 39] Nanotubes [24]

The electrochemical polymerization of aniline is the most common approach for PANI synthesis [22]. The main disadvantage of the electrochemical synthesis of PANI is the need to use an electrically conductive scaffold for the synthesis of polymer films [40]. The main advantage of this method is the high quality of the final

product containing a low level of impurities, that does not require further purification from unreacted monomer and initiator molecules. Table 2 shows the examples of MI-PANI synthesis via electrochemical polymerization methods in terms of the morphology of the resulting structures and the electrode materials.

Table 2

**Electrochemical synthesis of molecularly imprinted polyaniline**

MI-PANI structure	Synthesis method	Template	Electrode material	Ref.
Nanowires	CA, CV	Chloramphenicol	Gold	[32]
		Histamine		[41]
		Cardiac troponin T		Graphene
Film	CV	Azithromycin	Glassy carbon electrode	[42]
		Cefixime		[43]
		$\beta$ -Amyloid-42	Copper@carbon nanotubes	[44]
		<i>L</i> -ascorbic acid	Graphite	[45]
		n/a	Dapsone	Platinum with nanoparticles of Fe <sub>3</sub> O <sub>4</sub>
	PE	Melamine	Glassy carbon electrode	[35]
				[31]

Note. CA – Chronoamperometry, CV – Cyclic voltammetry, PE – Potentiodynamic electropolymerization.

According to the applied electrode voltage mode, three electrochemical polymerization reaction methods can be realized – cyclic voltammetry [21], differential voltammetry [46], and chronoamperometry [33]. Synthesis is carried out on the surface of electrodes made of an inert conducting material in aqueous solutions containing background

electrolytes and acids to increase ionic conductivity.

Electrochemical polymerization is mainly used for the synthesis of MI-PANI films on the electrode surface, and precise control of film thickness is possible. However, the synthesis of complex MI-PANI structures requires the use of special matrices [47]. In addition, obtaining MI-PANI in large quantities



via electrochemical polymerization is difficult, as the synthesis is only carried out on the area limited by the electrode surface.

Chemical polymerization of aniline is a simple method to produce PANI with different morphologies in large quantities [48]. The main advantages of chemical polymerization over the electrochemical method are the wide choice of monomers and the ability to synthesize the polymer matrix on any type of substrate or without substrates. Chemical polymerization of aniline is carried out in the presence of various oxidants [49] such as potassium dichromate, potassium permanganate, iron (III) chloride and others. The most widely used oxidant is ammonium persulfate [50], as it has good solubility in aqueous media and provides a high yield (~90 %) of products.

The method and conditions of aniline chemical polymerization leads to differences in the electro-mechanical, morphological, structural and physical properties of resulted PANI [51] and, therefore, MI-PANI properties. Chemical polymerization makes it possible to obtain MI-PANI in nanowires and films forms, similar to electro polymerization, as well

as a wide range of other MI-PANI nanostructures, including granules, nanotubes, microspheres, and nanospheres. MI-PANI nanostructures have several advantages over films, namely a large surface area and high porosity of particles that allows decreasing the cost of analysis and sample volume [52]. Moreover, the morphology of MI-PANI nanostructures facilitates the immobilization of various biocatalysts and bioreceptors on their surface, thereby increasing the sensitivity of analysis [53]. The wider linear range of analyte determination is obtained due to the large specific surface area of MI-PANI nanoparticles, while the low detection limit is attributed to the high electrical conductivity of PANI [15].

### Analytical Application of Molecularly Imprinted Polyaniline

MI-PANI has already found successful applications for analytical purposes. Its main purpose is modification of electrodes, as MI-PANI obtained by both electrochemical and chemical methods can be used for such purpose. Some examples of analytical systems based on MI-PANI are provided in Table 3.

Table 3

Analytical characteristics of molecularly imprinted polyaniline

MI-PANI structure	Template	LOD, $\mu\text{M}$ ( $\text{ng}\cdot\text{mL}^{-1}$ )	LR, $\mu\text{M}$ ( $\text{ng}\cdot\text{mL}^{-1}$ )	Ref.
Film	Adrenaline	0.001	0.001–100	[54]
	Azithromycin	$0.1 \cdot 10^{-3}$	$\{0.3\text{--}920\} \cdot 10^{-3}$	[43]*
	$\beta$ -Amyloid-42	(400)	(1–66)	[45]*
	Cardiac troponin T	$(8.0 \cdot 10^{-3})$	(0.02–0.09)	[21]*
		(40)	$(100\text{--}8 \cdot 10^3)$	[42]*
	Cefixime	$7.1 \cdot 10^{-3}$	0.02–0.95	[44]*
	Flucarbazon	5.8	$100\text{--}1 \cdot 10^5$	[55]*
	Glucose	$1.2 \cdot 10^3$	$\{2\text{--}11\} \cdot 10^3$	[22]
	Histamine	0.21	0.5–1000	[41]*
	L-ascorbic acid	1.0	1–100	[34]*
Melamine	$4.5 \cdot 10^{-4}$	$\{0.6\text{--}16\} \cdot 10^{-3}$	[31]*	
Nanotubes	Horseradish peroxidase	$(3.6 \cdot 10^{-4})$	$(10^{-3}\text{--}1 \cdot 10^2)$	[24]
		$(7 \cdot 10^{-2})$	(0.05–10)	[26]
Nanowires	Chloramphenicol	$1.0 \cdot 10^{-4}$	–	[32]*
		$1.2 \cdot 10^{-3}$	$10^{-2}\text{--}10^3$	[33]*
Nanofibers	Aldicarb	$5.0 \cdot 10^{-4}$	$\{50\text{--}80\} \cdot 10^{-3}$	[28]
	Calycosin	$8.5 \cdot 10^{-2}$	0.42–129	[29]*
Nanoparticles	Ovalbumin	$(1.0 \cdot 10^{-9})$	$(10^{-4}\text{--}1)$	[36]
	p-Nitrophenol	20	60–140	[37]
	Paracetamol	50	$0.4\text{--}1 \cdot 10^3$	[15]

\* – MI-PANI synthesized by electrochemical methods; LOD – Limit of detection; LR – Linear range.



As we can see, the MI-PANI can be used for determination of low- and high molecular weight compounds on the nanomolar concentrations. The MI-PANI nanoparticles (NPs) for such purpose were synthesized via oxidative polymerization of aniline within the micelles [15] with ammonium persulfate used as an oxidizing agent. A selective recognition element based on MI-PANI NPs has been developed for paracetamol determination; this element is characterized by a particularly low detection limit. The developed approach has several advantages: polymerization is carried out in an aqueous medium and is harmless to the environment; simultaneous synthesis of NPs and imprinting reduce the time of MIPs creation; the approach is universal and can be adapted for other template molecules.

Various nanocomposites can be used as scaffold for MI-PANI synthesis. For example, a binary  $\text{CuWO}_4$ @PANI nanocomposite has been used by Ponnaiah S.K. and Periakaruppan P. [30] to determine the quercetin level in blood, urine and natural samples without complicated pretreatment. Fatahi et al. have developed [56] an electrochemical sensor based on  $\text{Fe}_3\text{O}_4$ /PANI-Cu<sup>II</sup> microspheres for dexamethasone monitoring in real samples, such as human urine and serum using differential pulse voltammetry. The urine sample was centrifuged and diluted 10 times without any further pretreatment. The serum sample was treated with methanol to precipitate proteins, and precipitated proteins were subsequently separated out by centrifugation.

Quantum dots are also an interesting nano-dimension scaffold. Li et al. [57] have reported the application of CdTe quantum dots as a selective and sensitive fluorescent nanosensor based on surface imprinting technology. The sensor was used for evaluation of rutin in fruits, vegetables and medicinal plants in the concentration range of 0.1–30 mM, with the detection limit being 0.04 mM. Authors of study [58] describe a nanocomposite probe based on quenching the fluorescence of quantum dots to detect lomefloxacin. The efficiency of the described probe is based on a combination of the quantum dots sensitivity, the MIP selectivity, and the high adsorption PANI affinity.

In some cases, expensive scaffolds and equipment are not required for sensor fabrication. For example, Chen et al. [22] report the procedure for chemical synthesis of MI-PANI performed on the surface of paper strips, that were then connected to the electrode surface. This manufactured electrochemical sensor can determine the concentration of glucose in the blood. The authors noted lower temperature and humidity influence, simplicity, and low cost of such sensors compared to existing ones.

Saksena K. et al. [34] describe the development of an enantioselective sensor for chiral and quantitative monitoring of *L*-ascorbic acid in serum medium. Polymerization has been performed using cyclic voltammetry on the surface of the graphite pencil rod resulting in formation of a uniform, homogeneous and ultrathin film of MI-PANI.

Wang et al. [24] describe the facile horseradish peroxidase electrochemical biosensor based on modification of a glassy carbon electrode with a MI-PANI nanotubes chemically synthesized in aqueous solution. Recently, we have also obtained nanowire structured MI-PANI for horseradish peroxidase determination [26]. The developed approach has been used to determine the enzyme immobilized on the inner surface of glass polycapillary using optical detection via a chromogenic reaction with 3,3',5,5'-tetramethylbenzidine.

A large number of studies [32, 33, 35, 43, 58] is dedicated to the development of MI-PANI based antibiotic sensors. Compared to other means of antibiotic analysis, the use of molecular imprinting-based devices usually does not require a time-consuming stage of sample preparation. In addition, MIP based sensors are comparatively simple and cheap in manufacturing, have high selectivity and reproducibility, and can be used for electro-inactive compounds. The use of MI-PANI is not limited to antibiotics analysis in food and wastewaters, for example application of MI-PANI for clinically important substances has been reported by many studies [21, 22, 29, 31, 36, 42, 45, 46, 54].

## Conclusion

Therefore, we have shown that PANI is the great potential material for MIPs synthesis for low- and high-molecular weight-targets including complex objects. The obvious advantages of PANI as a matrix polymer for MIPs synthesis are high stability and biocompatibility. Various procedures of MI-PANI synthesis result in obtaining MIPs layers with dramatically different structure and properties. These considerations suggest that PANI will remain the object of keen interest in the field of molecular imprinting for a long time.

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