

# O ensino e a pesquisa em **QU** **MICA**

Cleiseano Emanuel da Silva Paniagua  
(Organizador)

# 2

  
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## APRESENTAÇÃO

O e-book: “O ensino e a pesquisa em química” volume II é constituído por quinze capítulos de livro que foram reunidos em três grandes áreas temáticas, a saber: *i)* química analítica: determinação, otimização e validação; *ii)* desenvolvimento de adsorventes e catalisadores para remoção de diferentes classes de contaminantes e aplicação industrial e *iii)* temas diversos.

A primeira temática é constituída por seis capítulos e apresentam diversos estudos, entre os quais: *i)* determinação quantitativa de glicazida em comprimidos e os problemas provenientes do uso de comprimidos pelo sistema de partição não homogêneo; *ii)* a determinação de Hidrocarbonetos Policíclicos Aromáticos (HPAs) empregando-se a técnica de voltametria; *iii)* a validação de um sistema fotocolorímetro em análises para o ensino de química na Universidade Tecnológica de Gutiérrez Zamora na cidade de Vera Cruz no México; *iv)* a importância da otimização para melhor entendimento dos estudos cinéticos em uma reação de hidrologenação; *v)* a triagem fitoquímica e análise de propriedades antioxidantes e *vi)* avaliação de estruturas metálicas orgânicas como fase estacionária em Cromatografia Líquida de Alta Eficiência (HPLC).

A segunda temática é composta por cinco capítulos e apresentam: estudo de revisão que demonstram: *i)* o potencial de extração de fósforo em efluentes líquidos; *ii)* estudo de Montmorilonita como potencial adsorvente e aplicação em sistemas de fluxo contínuo e *iii)* avaliação e estudo de diferentes catalisadores para remoção de inúmeras classes de poluentes em matrizes aquáticas e reforma do etanol com vapor d’água.

Por fim, a terceira temática que apresenta quatro diferentes estudos que contemplam a corrosão obtida por pulverização de gás frio, a importância e utilização de supressores de poeira na mineração, preparação de nanopartículas poliméricas enriquecidas com óleos essenciais poliméricas e estudo de revisão das propriedades químicas da série de lantanídeos.

Nesta perspectiva, a Atena Editora vem trabalhando por meio do incentivo de publicações de trabalhos de pesquisadores de todas as regiões do Brasil e de outros países com o intuito de colaborar com a publicação de e-books e, conseqüentemente, sua divulgação de forma gratuita em diferentes plataformas digitais de fácil acesso. Logo, a Atena Editora contribui para a divulgação e disseminação do conhecimento científico gerado dentro de instituições de ensino e pesquisa e que pode ser acessado de qualquer lugar e em tempo real por qualquer pessoa interessada na busca pelo conhecimento.

Cleiseano Emanuel da Silva Paniagua


## SUMÁRIO

### **CAPÍTULO 1..... 1**

#### **DETERMINAÇÃO QUANTITATIVA DE GLICLAZIDA EM COMPRIMIDOS SULCADOS**

Jacqueline Cristinne Guimarães Vidal

Maria Amélia Albergaria Estrela

 <https://doi.org/10.22533/at.ed.2352131081>

### **CAPÍTULO 2..... 13**

#### **DETERMINAÇÃO VOLTAMÉTRICA DE HIDROCARBONETOS POLICÍCLICOS AROMÁTICOS USANDO UM ELETRODO DE PASTA DE CARBONO MODIFICADO COM FTALOCIANINA DE ZINCO**

Daniel Jackson Estevam da Costa

Rhayane Silva Rodrigues do Nascimento

Larissa da Silva Pereira

Janete Clair da Silva Santos


Camila Luciana Silva de Mesquita

Fátima Aparecida Castriani Sanches-Brandão

William Farias Ribeiro

Francisco Antonio da Silva Cunha

Mário César Ugulino de Araújo

 <https://doi.org/10.22533/at.ed.2352131082>

### **CAPÍTULO 3..... 25**

#### **VALIDACIÓN DE UN FOTOCOLORÍMETRO PARA ANÁLISIS CUANTITATIVOS EN LA ENSEÑANZA DE LA QUÍMICA**

Raúl Alejandro Limón Hernández

Verónica López Hernández


Fidel Alejandro Aguilar Aguilar

Iriana Hernández Martínez

José Luis Xochihua Juan

Arsenio Sosa Fomperosa

Oscar Enrique Morales Moguel

 <https://doi.org/10.22533/at.ed.2352131083>

### **CAPÍTULO 4..... 40**

#### **OTIMIZAÇÃO NUMÉRICA E ESTUDO CINÉTICO DA REAÇÃO DE HIDROALOGENAÇÃO DO ALFA-PINENO**

Vladimir Lavayen

Thágor Moreira Klein

Chádia Schissler

Leticia Antunes Natividade

Alexandre Chagas

Jacqueline Arguello da Silva

 <https://doi.org/10.22533/at.ed.2352131084>

**CAPÍTULO 5..... 50**

TRIAGEM FITOQUÍMICA E ANÁLISE QUALITATIVA DO POTENCIAL ANTIOXIDANTE DOS EXTRATOS FOLIARES DE *Cinnamomum zeylanicum* E *Cinnamomum burmannii*

Ana Francisca Gomes da Silva

Valéria Flávia Batista da Silva

Carolina Lopes

 <https://doi.org/10.22533/at.ed.2352131085>

**CAPÍTULO 6..... 58**

MOFs (METAL ORGANIC FRAMEWORKS) AS A STATIONARY PHASE IN LIQUID CHROMATOGRAPHY (HPLC)


Tamires dos Reis Menezes

Kátilla Monique Costa Santos

Sílvia Maria Egues Dariva

Juliana Faccin de Conto Borges

Cesar Costapinto Santana

 <https://doi.org/10.22533/at.ed.2352131086>

**CAPÍTULO 7..... 76**

POTENCIAL DE EXTRAÇÃO DE FÓSFORO DE EFLUENTES LÍQUIDOS – REVISÃO DE LITERATURA

Luciana Faria Caetano de Souza

Suzana Maria Loures de Oliveira Marcionilio

Ana Carolina Ribeiro Aguiar

Ana Paula Cardoso Gomide

 <https://doi.org/10.22533/at.ed.2352131087>


**CAPÍTULO 8..... 84**

ESTUDO DE OBTENÇÃO DE MONTMORILONITA POR PRECIPITAÇÃO SEQUENCIAL PARA USO COMO ADSORVENTE PARA SISTEMAS DE FLUXO CONTÍNUO

Kathely Priscila de Souza Trindade

Graciele Vieira Barbosa

Alberto Adriano Cavalheiro

 <https://doi.org/10.22533/at.ed.2352131088>

**CAPÍTULO 9..... 97**

OXIDAÇÃO CATALÍTICA DO CORANTE AZUL DE METILENO USANDO NOVOS CATALISADORES  $Fe_2O_3$


Matheus de Araújo Moura

Henrique Rebouças Marques Santos

Márcio Souza Santos

Rennan Noronha de Franca

Alexilda Oliveira de Souza

 <https://doi.org/10.22533/at.ed.2352131089>

**CAPÍTULO 10..... 107**

**AVALIAÇÃO DA SELETIVIDADE DO CATALISADOR FORMADO A PARTIR DO SULFATO DE MAGNÉSIO *in situ* NA PIRÓLISE CATALÍTICA DA BIOMASSA DE MACRÓFITA AQUÁTICA PARA OBTENÇÃO DE BIO-ÓLEO PIROLÍTICO**


Júnior da Silva Camargo  
Sonia Tomie Tanimoto  
Fernando Alves Ferreira  
Vitor Vinícius Anjos Bonfim Ribeiro  
Regineide de Oliveira Lima  
Ricardo Faustino Rits de Barros  
José Alfredo Valverde  
Layssa Aline Okamura  
Paulo Renato dos Santos

 <https://doi.org/10.22533/at.ed.23521310810>

**CAPÍTULO 11 ..... 120**

**REFORMA DO ETANOL COM VAPOR D' ÁGUA: AVALIAÇÃO DA ATIVAÇÃO PRÉVIA DO CATALISADOR Cu/Ni/Na<sub>2</sub>O-Nb<sub>2</sub>O<sub>5</sub>**


Laura dos Santos Costa  
Maria Eduarda Bogado dos Santos  
Marília de Oliveira Camargo  
Marcos de Souza  
Isabela Dancini Pontes

 <https://doi.org/10.22533/at.ed.23521310811>

**CAPÍTULO 12..... 129**

**STUDY OF CORROSION AND MECHANISMS OF COATINGS OBTAINED BY COLD GAS SPRAY USING OPEN CIRCUIT POTENTIAL AND MICROSCOPIC ANALYSIS**

Fernando Santos da Silva  
Sergi Dosta  
Assis Vicente Benedetti

 <https://doi.org/10.22533/at.ed.23521310812>

**CAPÍTULO 13..... 144**

**SUPRESSORES DE POEIRA DE MINERAÇÃO**

Stéphane Miranda Francisco  
Enio Nazaré de Oliveira Júnior  
Ana Maria de Oliveira


 <https://doi.org/10.22533/at.ed.23521310813>

**CAPÍTULO 14..... 156**

**PREPARATION OF SUB-50 NM POLYMERIC NANOPARTICLES LOADED WITH ESSENTIAL OILS**

Hened Saade Caballero

 <https://doi.org/10.22533/at.ed.23521310814>

<b>CAPÍTULO 15.....</b>	<b>160</b>
QUÍMICA DE LANTANÍDEOS	
Jorge Fernando Silva de Menezes	
Rodrigo Galvão dos Santos	
 <a href="https://doi.org/10.22533/at.ed.23521310815">https://doi.org/10.22533/at.ed.23521310815</a>	
<b>SOBRE O ORGANIZADOR.....</b>	<b>192</b>
<b>ÍNDICE REMISSIVO.....</b>	<b>193</b>

## MOFs (METAL ORGANIC FRAMEWORKS) AS A STATIONARY PHASE IN LIQUID CHROMATOGRAPHY (HPLC)

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**ABSTRACT:** The MOFs belong to a class of coordination polymers that can be used as micro and meso porous materials of high-capacity

adsorption. These materials combine inorganic parts (metal ions) with organic parts (binders) creating three-dimensional crystalline structures with covalent bonds. The peculiar characteristics (high porosity, selectivity, multiple active sites) make the MOFs a promisor adsorbent as a new stationary phase of high performance for liquid chromatography. This work is an exploratory study of published articles in the literature in several scientific journals, between 2008 and 2018, in order to create summary and recommendations of experimental results regarding to MOF applications as a stationary phase in liquid chromatography (HPLC). In order to overcome the challenge of loss of column efficiency due to the irregular sizes and shapes of the particles, there is an absolute growth in pre-treatment and modifications area of the MOFs that aims to improve the efficiency and activity.

**KEYWORDS:** MOFs, ZIFs, liquid chromatography, HPLC.

## 1 | INTRODUCTION

The separation process for obtaining pure substances is of crucial importance for the chemical and allied industries. In this context, the chromatographic processes stand out. High-performance liquid chromatography (HPLC) is among the most widely used as a tool in both biochemistry and separation analyzes (HAYES et al., 2014).

Chromatography can be described as a physicochemical method of separating components from a mixture, accomplished by

distributing these components into two phases, which are in contact. One phase remains stationary while the other moves through it. During the contact of the mobile phase on the stationary phase, the components of the mixture are distributed over the two phases so that each phase is selectively retained by the stationary phase, resulting in different retention times of these components (COLLINS et al., 2006).

The silica stands out as the most used material in the processes with HPLC, however, new porous materials emerge as a possibility to compose the stationary phase, as the coordination polymers.

A new class of microporous coordination polymers appears as an alternative to HPLC stationary phase are the so-called MOFs (Metal-Organic Frameworks). Metallurgical networks consist of clusters or metal ions coordinated to organic binders containing potential voids. The recent interest in these materials is a consequence of the combination of important characteristics such as crystallinity, porosity and the existence of strong metal-binder interaction. This unique combination of properties elevates MOFs to a special class of adsorbents as it conducts materials with greater thermal stability and greater mechanical stability. In this way, the MOFs are materials with great potential of adsorption and chromatography applications, besides others such as catalysis and controlled release of drugs (AHMAD et al., 2009; NORDIN et al., 2015).

The synthesis and development of MOFs are relatively recent, with the consolidation of the pioneering work of the so-called reticular chemistry in the late 1990s (YAGHI, 1999). Materials and methods for the preparation of materials with significant chemical, mechanical and nanoporosity robustness emerged exponentially in the later decades (LONG and YAGUI, 2009; LI et al., 2009). These adsorbents exhibit a great potential of application, especially in the manufacture of chemical products, separation processes, catalysts among others, in the area of fine chemistry. Due mainly to the possibility of modulation of the porous structure and the electronic properties of the active sites. The porosities of the MOFs are located between the mesoporous zeolites and the mesoporous silicates, being considered as nanostructured materials (KINIC et al., 2017).

Another class of MOFs that presents a high interest are the ZIFs (Zeolite Imidazole Frameworks), since these materials present high thermal and chemical stability of zeolites, with the specific advantages of MOFs as high surface area, Zeolite Imidazole Frameworks). ZIFs are porous and crystalline networks obtained from zeolites in which the metal ions are bound to the backbone by imidazole units (ZHU et al., 2017).

In particular, the possibility of post-synthesis modification of these MOFs, from the change of the organic part, promotes different chemical environments and can serve as a reaction medium with control of chemical properties. It thus becomes a promising class of adsorbent materials. The peculiar characteristics such as high porosity, shape selectivity, and multiple active sites make the MOFs a promising adsorbent as a new high-performance stationary phase for liquid chromatography (EHLING et al., 2018).



The literature-based approaches based on MOFs are very important for knowledge of the ability of MOFs in the adsorption/separation process and for better knowledge of the material, obtaining good results due to high adsorption rates (ZHANG and CHEN, 2017).

Since the advent of this new material the investigations have focused mainly on the adsorption to a gas phase, however, some works dealing with MOFs for separations of liquid phases have been receiving a lot of attention. Three interesting reviews have been published (Vororde et al., 2014, ZHANG et al., 2017 and MUKHERJEE et al., 2018) containing the MOF studies for liquid phase separations. MUKHERJEE and collaborators also focus on their study on the pore flexibility of metal-organic structures in the face of the evolution of studies already published in the literature.

In this context, the objective of this review was to conduct an exploratory study of the evolution of the articles published in the literature, concerning the applications of MOFs as a stationary phase in high performance liquid chromatography (HPLC), with the aim of highlighting the evolution of these stationary phases, from the use of the pure, as well as, in the supports made to overcome the problem of efficiency loss in the column due to the non-uniformity of the particles. The abstract has articles published between the years 2008 and the present year, 2018. Initial surveys identified that in the period prior to 2008 there is a shortage of articles that describe in greater detail.

## 2 | EXPLORATORY STUDY

### 2.1 MOF in Liquid Chromatography

Cychoosz et al. (2008), although they did not perform a work using the liquid chromatography technique, developed a study based on the liquid phase adsorption by microporous coordination polymers (MCPs), MOFs and subfamily of MOFs, to remove organosulfide compounds that later served as a basis for several other studies. MCPs exhibit superior adsorption capacity when compared to activated carbon and zeolite, but the quantitative determination on adsorption of large molecules by MCPs (Microporous coordination polymers) is still poorly exploited, although they exhibit adequate pore size.

In the work, 5 types of MCPs (UMCM-150, MOF-505, HKUST-1, MOF-5 and MOF-177) were studied for the adsorption of benzothiophene, dibenzothiophene, and 4,6-dimethyldibenzothiophene from isooctane solutions. The choice of the MCPs was an attempt to ascertain different structures with different pore sizes and different metal clusters. Saturation was not achieved for all the isotherms studied in the work, indicating that these materials have a potential for even greater amounts of adsorption, Figure 1. Another factor that attracted a lot of attention in the study was the comparison of the capacities of these materials in relation to reference zeolite, Na (Y). For comparison, the adsorption capacities were measured and MOF structures were adsorbed 5 to 6 times

more than the reference zeolite.

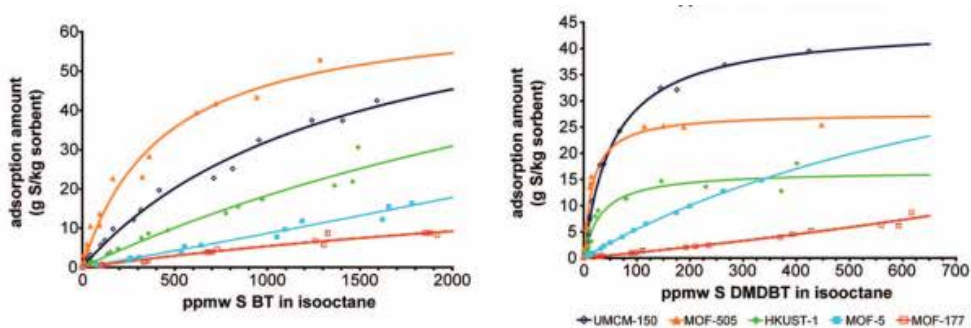


Figure 1. Adsorbent isotherms of benzothiophene and 4,6-dimethyldibenzothiophene in isooctane solutions with the MCPs: UMCM-150, MOF-505, HKUST-1, MOF-5 e MOF-177. Reprinted (adapted) with permission from (CYCHOSZ *et al.*, 2008). Copyright American Chemical Society.

The work of ALAERTS *et al.* (2008) established how essential it is for the application of MOFs that they undergo an activation process being pretreated to obtain a larger volume of pores because during the process of synthesis or modification some organic binders or even non-volatile solvents can lodge in the pores.

Alaerts and colleagues proposed the total removal of the terephthalic acid housed in the pores of MOF MIL-47 during the calcination process that was performed at 573K, suggesting that there would be greater accessibility by increasing the pore volume and, consequently, maximizing the adsorption rate of xylenes, reporting that this step is essential to apply the MOFs to selective separations in the liquid phase. They also performed pulse and breakthrough chromatographic experiments using a stainless steel 5 cm long and 4 mm internal diameter filled with MIL-47 using hexane as the mobile phase. It was noted, then, that the temperature increases both completely removed the terephthalic acid from the pores, as it prevented the oxidation of the MIL-47 before pretreatment. However, under the activation conditions, it was not possible to avoid oxidation. The application of MIL-47 to the separation was impaired since the acid removed contributes in the selectivity between p-xylene and m-xylene. However, the activated MIL-47 is quite selective for separations of para-meta-mixtures of aromatics like ethyl toluene, xylenes, dichlorobenzenes, toluidines, and cresols.

The activation process of the MOFs, conditioned for a certain time and a high temperature, which varies with the type of MOF according to the temperature that the structure begins to degrade or collapse, has been carried out as a standard pretreatment of the material prior to characterizations and / or applications in both liquid adsorption and gas adsorption studies to obtain greater pore access and better adsorption rates.

Ahmad points out that analyzing previous work such as Cychosz's, MCPs, MOFs, offer an attractive alternative to other materials already used as a stationary phase

because of their adjustable pore sizes, high surface area and commercial availability. It thus becomes an excellent material with high potential for adsorption and high selectivity for liquid chromatography. Then, a study was carried out with coordinated polymers of microporous polymers derived from carboxylates coordinated with copper (HKUST-1) and zinc (MOF-5), two of the materials studied by Cychosz. The two materials were packed on a HPLC column and hexane and dichloromethane were used as the mobile phase for the selective separation of organic compounds including; benzene, ethylbenzene, styrene, naphthalene, anthracene, phenanthrene, pyrene, 1,3,5-triphenylbenzene and 1,3,5-tris(4-bromophenyl) benzene and, in most cases, excellent separation with different retention and good adsorption rate, Figure 2 and 3. However, it was observed a high counter pressure and the existence of extended peaks.

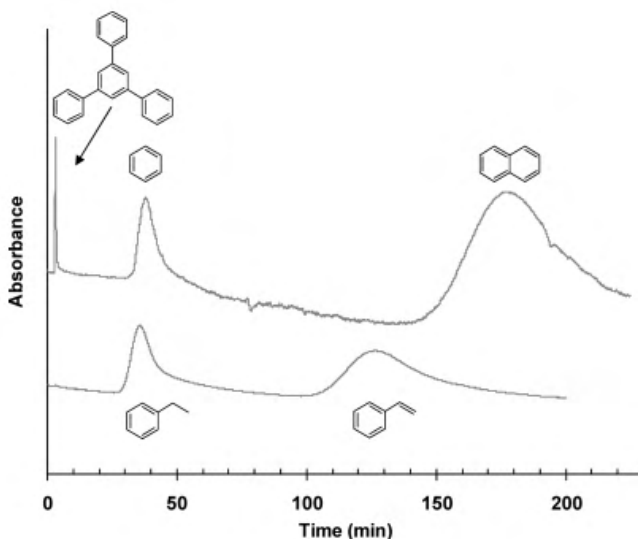


Figure 2. Chromatogram of a mixture of 1,3,5-triphenylbenzene, benzene and naphthalene. Chromatogram of styrene and ethylbenzene. Reprinted (adapted) with permission from (AHMAD *et al.* 2009). Copyright American Chemical Society.

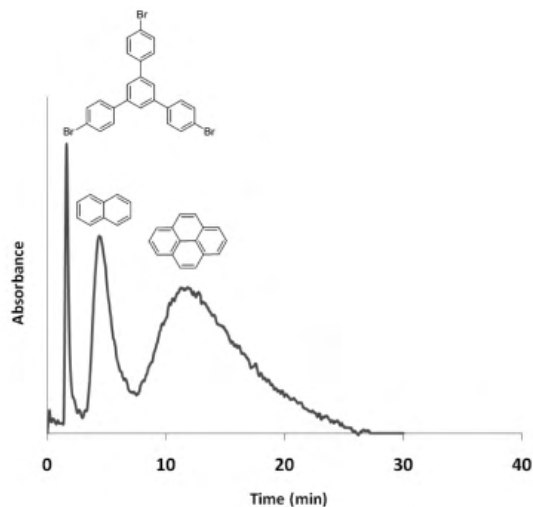


Figure 3. Chromatogram of 1,3,5-tris(4-bromophenyl) benzene, naphthalene and pyrene using MOF-5 as stationary phase. Reprinted (adapted) with permission from (AHMAD *et al.* 2009). Copyright American Chemical Society).

Despite the several positive characteristics of MOFs, such as high adsorption rates and efficiency in the separation of several substances such as aromatic compounds studied by Ahmad, it is evident that the wide distribution of particle sizes and irregular shapes can generate against pressure and cause a decrease in efficiency of the HPLC columns filled with the MOF, as can be observed in Ahmad's work where peak amplification and low intensity occurred. Faced with this problem, after the year of 2009, some studies appeared seeking alternatives to minimize it and improve the efficiency of columns containing the stationary phase.

One of the alternatives to reduce separation time and improve column efficiency is to manufacture silica-MOF composites for fast and efficient HPLC separation, as can be seen in the work of Ameloot *et al.*, By comparing the two columns, MOF and Silica-MOF. Although this manufacture of silica-MOF composites is highly relevant and promising, this activity requires great challenges.

AMELOOT *et al.* (2010) studied silica-MOF as a stationary phase in HPLC. The work consists in introducing a method based on a clear precursor solution that allows the formation of MOF within the pores of silica granules, often used in chromatography, leading to spheres with a uniform particle size of 3  $\mu\text{m}$ . Silica was modified with MOF, [Cu<sub>3</sub> (BTC)<sub>2</sub>]. In the study, we performed column tests containing MOF, [Cu<sub>3</sub> (BTC)<sub>2</sub>], unmodified silica and silica modified with MOF. They used heptane as the mobile phase and a column 15 cm long and 4.6 mm in diameter. As a result, the columns containing [Cu<sub>3</sub> (BTC)<sub>2</sub>] showed high backpressure (around 200 bar) and fairly broad peaks. The column containing the unmodified silica despite having a lower counter-pressure (around 54 bar, could not

separate the ethylbenzene-styrene mixture.) However, the column containing the silica-MOF compound was able to separate the mixture of ethylbenzene and styrene effectively with lower retention time, lower back pressure (around 47 bar) with intense and narrow peaks, Figure 4.

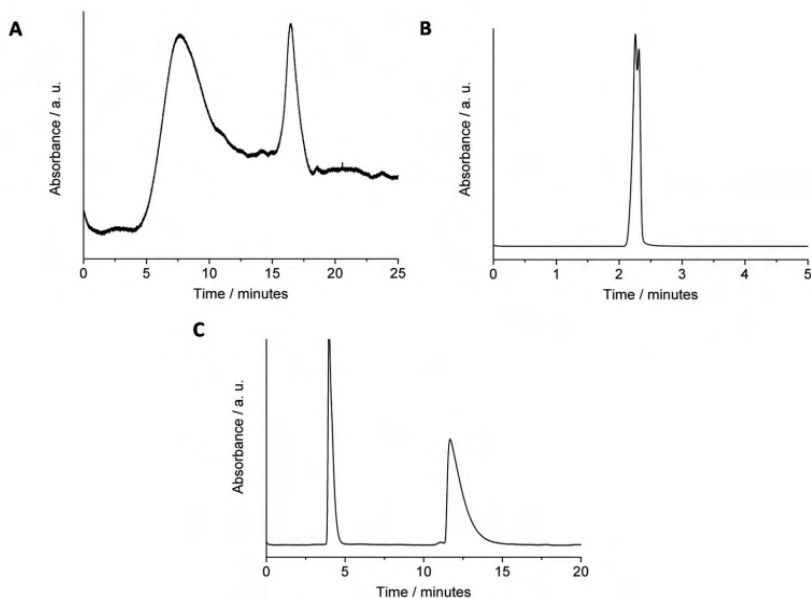


Figure 4. (A) Injection of a mixture of ethylbenzene styrene into a pure [Cu<sub>3</sub> (BTC) <sub>2</sub>] column. (B) Silica column without modification. (C) Silica column modified with [Cu<sub>3</sub> (BTC) <sub>2</sub>]. Reprinted (adapted) with permission from (AMELOOT *et al.*, 2010). Copyright American Chemical Society.

Aware of the positive characteristics of the MOFs and knowing the challenge that the irregular sizes and shapes of the particles can cause in an HPLC column, Fu *et al.* (2013) they studied a new possibility, using a subclass of MOFs called ZIFs. Among ZIFs, ZIF-8 was chosen for loading the structural advantages of MOFs and features present in the compounds as zeolites as high surface areas, high chemical and mechanical stability. They used ZIF-silica to separate EDCs (bisphenol A (BPA), b-estradiol (E2) and p- (tert-octyl) phenol) and pesticides (thiamethoxam, hexaflumuron, chlorantraniliprole and pymetrozine) and named the ZIF -8 @ SiO<sub>2</sub> for this stationary phase. They obtained a rapid separation of EDCs and pesticides with low column backpressure. We have also demonstrated that ZIF-8 is effective for separating the large molecules tested because of the surface interactions between them and the analytes. This study offers the possibility of preparation of multifunctional composites based on large applications of MOFs in HPLC separation.

In addition, Fu *et al.* (2013) studied the effect of ZIF-8 density by testing the influence of different layers of the carrier, silica, on HPLC separation, which they called the number of growth cycles of ZIF-8. They found that the density was controlled by the number of growth

cycles of ZIF-8. Resolutions of EDCs and pesticides were clearly improved and baseline separation was achieved in the column packed with ZIF-8 @ SiO<sub>2</sub> prepared after three cycles of ZIF-8 bark growth, figure 5.

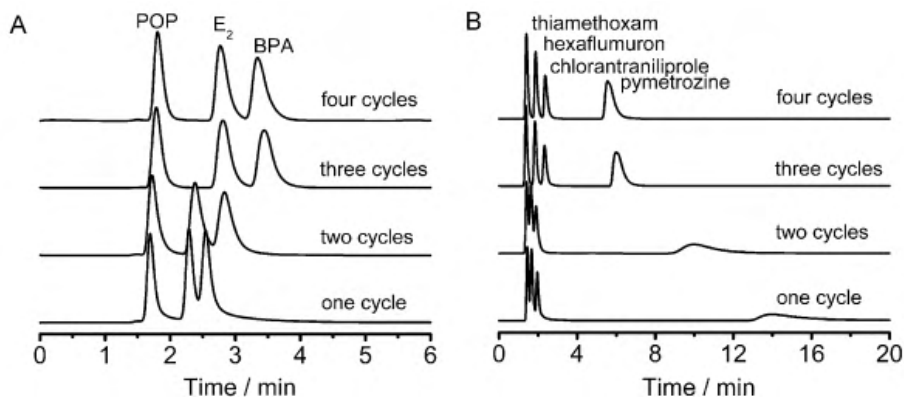


Figure 5. Chromatograms on ZIF-8 @ SiO<sub>2</sub> columns with different numbers of growth cycles. (A) EDCs (B) pesticides. Reprinted (adapted) with permission from (FU *et al.*, 2013). Copyright American Chemical Society.

QIN *et al.* (2015), in his study, reported that there are few data on MOFs structures, one reason being the heterogeneity of the material with respect to particle size and morphology, making it difficult to accurately describe kinetic data. They demonstrate the manufacture of liquid phase epitaxy (LPE) MOFs with functional carboxylic groups on the nuclear surface and bind the first layer of MOF. The epitaxy process can produce, in support models, metallic-organic structures attached to the thinner surface, which is more ordered, oriented and homogeneous. Figure 6 (SURMOFs, surface attached metal-organic framework thin films).

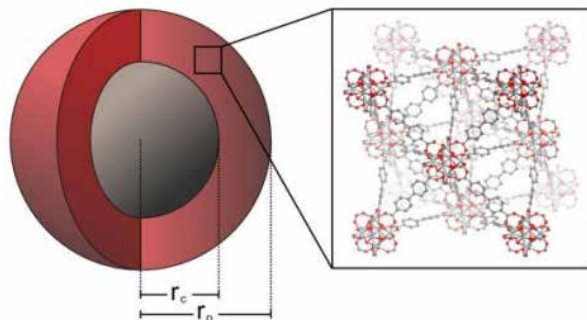


Figure 6. Schematic structure of the envelope of UiO-67 grown in carboxyl functionalized magnetic microparts:  $r_c$  refers to the radius of the magnetic particle and  $r_p$  defines the radius of the particle, including the shell. The structure of UiO-67 contains octahedral and tetrahedral pores of shared face. Reprinted (adapted) with permission from (QIN *et al.*, 2015). Copyright American Chemical Society.

Among the family of MOFs, the UiO series (University of Oslo) are of special interest because of their excellent thermal and chemical stability of the zirconium series UiO-67. The tests performed with the MOF in question were compared with simulations made from the ChromX software, developed for chromatographic modeling by the Institute of process Engineering in Life Sciences Section IV: Biomolecular Separation Engineering of the Karlsruhe Institute of Technology, in 2012. The experiments in HPLC were performed with ACN (acetonitrile) and water as the mobile phase on a 200 mm long and 1 mm internal diameter column.

Using phenolic derivatives (2,6-dimethylphenol, DMP, Benzene-1,3-diol, BZD and 2,6-dichlorophenol, DCP) to analyze kinetic properties in chromatographic experiments, it was noted that maximum loading capacity remained constant, however, the affinities of the studied molecules varied and showed a strong dependence of the applied solvent, ACN / H<sub>2</sub>O. The result was that the pore diffusivity was in the order of two or three times less than the diffusivity of the activated carbon. The results also indicate that, despite the simulations using a common diffusivity model, with the MOFs, the mass transfer is better elaborated, with surface diffusion effects and size effects exclusion.

In 2016, Lv et al. Used high performance liquid chromatography (HPLC) with fluorescence detector to determine thiols (organosulfur) compounds from waste water samples with MOF-5 prepared and used as a DSPE adsorbent for selective adsorption of wastewater sample. Chromatography experiments were performed using a 150 mm long and 4.6 mm diameter column and the mobile phase was composed of acetonitrile. In this work, a new carbazole-9-ethyl-2-maleimide (CAEM) reagent was also synthesized to improve the efficiency of MOF-5 and to increase its selectivity as the novel thio fluorescent derivatization reagent was designed and synthesized based on a reaction between maleimide and thiol groups. However, (CAEM) had no fluorescence, obtaining a fast reactivity and high selectivity for thiol group under mild conditions. The application of (CAEM) and MOF-5 resulted in selectivity advantages for low-limit detection thiols compared to other methods exhibiting excellent separation efficiency, Figure 7.

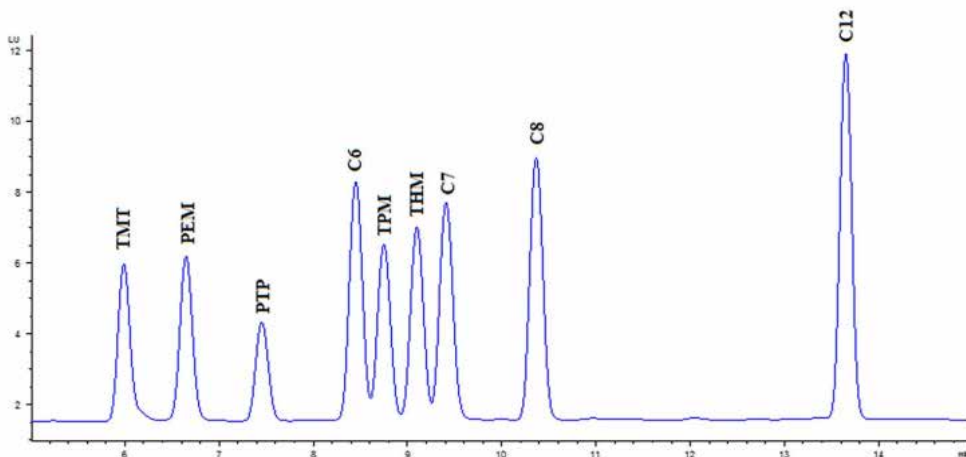


Figure 7. HPLC chromatogram of thiol derivatized: TPM: 4-(tert-butyl) phenyl methanethiol; PEM: 2-Phenylethylmercaptan; THM: Triphenylmethyl mercaptan; PTP: 2-Phenylthiophenol; TMT: 2-Thienylmethanethiol; C6: 1-Hexanethiol; C7: Heptyl mercaptan; C8: n-Octyl mercaptan; C12: Lauryl mercaptan. Reprinted (adapted) with permission from (LV *et al.*, 2016). Copyright American Chemical Society.

As already reported in the literature in previous years, despite the specific qualities of MOFs such as high surface area, crystallinity and large porosity, irregular shapes and wide particle size distribution hampered the separation efficiency and consequently increased backpressure in the HPLC column. In 2017 Qu and collaborators aiming to overcome this problem and obtain a larger pore volume and thus improve separation efficiency, have developed a new kinetic control method to deposit on the surface of the imidazolate structure (ZIF-8) shell-like silica beads as Figure 8, because small pores lead to slow mass transfer of analytes into the pores resulting in poor HPLC separation performance. Based on the above analysis, they proposed that if MOFs could be formed on the pore surface and not on the outer surface of the silica core, and with layers thin enough to make the original pore structure unaffected, the size of the pores of the obtained MOF beads would still be sufficiently large for HPLC separation and pore size deficiency could be overcome.



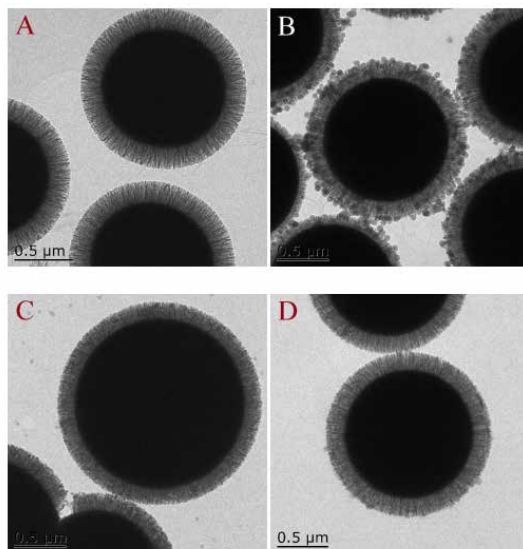


Figure 8. TEM images of SiO<sub>2</sub> @ dSiO<sub>2</sub> (A) SiO<sub>2</sub> @ dSiO<sub>2</sub>-ZIF-8 using different mixtures of DMF-methanol as solvent. The volume ratios of DMF: methanol were: B 5.3: 14.7; C 5.6: 14.4; D 5.8: 14.2. Reprinted (adapted) with permission from (QU *et al.*, 2017). Copyright American Chemical Society.

In the HPLC experiments, a 50 mm long, 4.6 mm diameter column with a mobile phase composed of ACN was used for the separation of xylene isomers. Thus, this study resulted in the obtaining of composite particles maintaining the silica bead macrostructure with efficient separation performance, but also showed the microporosity, high surface area and adsorption properties of MOFs. Spheres with dendritic shells (SiO<sub>2</sub> @ dSiO<sub>2</sub>) were used as a support, obtaining optimum yield and efficiency. It can be used to fill columns with the intention of maintaining the high level of separation.

In the same year, CHEN *et al.* (2017) have reported on the need to obtain pure m-p-DVB (divinylbenzene) as one of the essential constituents in the preparation of crosslinked styrene polymers used in the manufacture of ion exchange resins. In the knowledge of previous studies such as Yan *et al.* (1985) that showed the advantages of MOF MIL-53 (Fe) versus MIL-53 (Al, Cr) for the xylene, chlorotoluene and dichlorobenzene separations and FU *et al.* (2013) where aromatic compounds were separated using MIL-100 (Fe), CHEN *et al.* (DVB) and ethylvinylbenzene (EVB) isomers using MOFs, MIL-53 (Fe) and MIL-100 (Fe) as the stationary phase in HPLC with hexane as the mobile phase. In conclusion, they explained that separation was achieved in both columns, but the column with MIL-100 (Fe) provided a better separation of the DVB and EVB isomers than the MIL-53 (Fe) column. They also reported that the separation was not sufficient, showed short retention times and proposed that the particle uniformity should be increased to improve column efficiency.

In order to determine six pollutants of natural and residual waters (one polycyclic aromatic hydrocarbon (fluorene), two hormones (progesterone and estrone), two drugs

(carbamazepine and triclosan) and one disinfectant (atrazine)). In addition, the MOF-1, MOF-5 (Zn), MIL53 (Al), UiO-64 and MOF-74 (Zn) monomers were synthesized, characterized and applied by ROCÍ-BAUTISTA et al. miniaturized solid-phase extraction method under dispersive mode (D- $\mu$ SPE) optimized with high-performance liquid chromatography (HPLC). These pollutants have different natures and were selected because of the hydrophobicity, polarity, and aromaticity of each. The MOFs were chosen according to the versatility, extraction capacity, metallic nature, structure, and pore size. During the processes, it has been noted how difficult it is to predict how much each MOF will adsorb. The pore size and aperture, the presence of unsaturated metals and the nature of the metal have much influence on efficiency. However, MIL-53 (Al) proved to be the most suitable MOF in this case, obtaining the best selectivity, the methods were optimized and validated with this material.

In the same year, EHRLING et al. (2018) have challenged the relevant problems of industrial separation, such as the separation of aromatic isomers. With the knowledge that high performance liquid chromatography (HPLC) is the most used technique to combat these challenges, there is a need to find more efficient adsorbents. It also points out that the potential of the MOFs in HPLC had already been studied, however, they were used as a crystalline powder, resulting in high backpressure and low column efficiency. To do so, they proposed a study using silica and MOF in an attempt to combine the good silica packaging characteristics and the specific influence of the MOF material used.

In the study, they functionalized mesoporous silica beads with two chemically different structures with variable porosity, MIL-101 (polar) and UiO-67 (non-polar), to be used as a stationary phase in HPLC columns for the separation of C8 isomers, dichlorobenzene isomers, styrene and ethylbenzene. The method proposed in the work was layer-by-layer (lbl), thin films. They found from the SEM and the PXRD that the composites obtained after 20 cycles for MIL-101 @ SiO<sub>2</sub> and 18 cycles for UiO-67 @ SiO<sub>2</sub> obtained the best degree of functionalization of the surface being homogeneously coated and presented micro and mesoporosity, Figure 9.

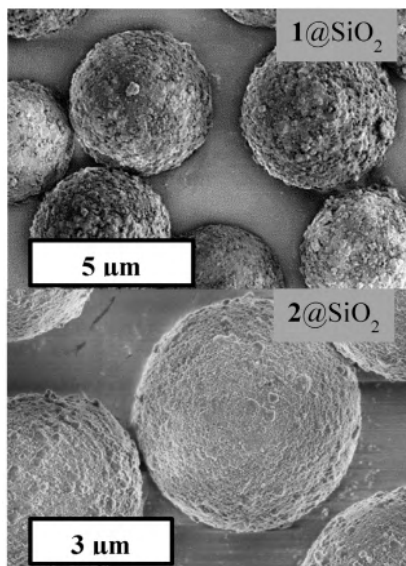


Figure 9. SEM images of MIL-101 @ SiO<sub>2</sub> structures (20 layer-by-layer cycles) and UiO-67 @ SiO<sub>2</sub> (18 layer-by-layer cycles). Reported as 1 SiO<sub>2</sub> and 2 SiO<sub>2</sub>, respectively. Reprinted (adapted) with permission from (EHLING *et al.*, 2018). Copyright American Chemical Society.

After application of the new stationary phase, it was observed that the silica / MOF composites showed higher affinity with the ortho-substituted analytes indicating that the polarity of the analytes directly influences the selectivity. Another important point is the influence of the solvent used to dilute the analytes, proving at work that small amounts of polar solvent can greatly reduce the separation efficiency.

Advances in the studies using liquid chromatography using MOFs as stationary phase are notorious and quite expressive. Within this context investigations for separations of chiral compounds appear. The chiral stationary phases behave analogously the stationary phases for non-chiral chromatographic separations, however, there is a need to use in these phases an optically active material or to produce a phase known as chiral MOF that is selective for the chiral compounds which are want to sort. Studies using MOFs for this purpose are quite recent and face major challenges as there is a clear need to find chiral stationary phases that are quite selective for a wide range of materials.

## 2.2 MOF em Cromatografia Líquida para separações quirais

Tanaka *et al.* (2012) drew attention to the homochiral separations, are the chromatographic separations of enantiomers. Only one article in 2007 would have addressed this issue. In the study, they reported a new homochiral compound synthesized from a mixture of (R) -H<sub>2</sub>BDA, Cu (NO<sub>3</sub>)<sub>2</sub> and monodisperse spherical silica gel, as a new stationary HPLC for the enantiomeric separation of various sulphoxides, also known as phase stationary. Two eluents were evaluated: hexane-EtOH (50/50) and one less polar,

hexane-i-PrOH (90/10). Nine sulfoxides (2,3,4,5,7,9,10,11,12) were completely separated from their corresponding enantiomers using the first eluent as the mobile phase. Two (14 and 16) were separated using the less polar eluent. However, the sulfoxides (6,8,13,15 and 17) did not show good separation in both solvents. This work may serve as a relevant basis for others in the future in enantioselective separation.

In 2014 Zhang's research group reported that although there were some papers bringing MOF studies for chiral separation, they still had a low enantioselectivity range and the irregular particle size was high and thus difficult to collect. In the study, they presented a chiral helical MOF [(CH<sub>3</sub>)<sub>2</sub>NH<sub>2</sub>][Cd (bpdc) 1.5], tested to separate several racemates as observed in Figure 10. They also studied the effect of temperature on retention and selectivity, since it is known that influence directly on most analytes. They realized that with increasing temperature the retention time decreased showing that the process was exothermic. As a result, they reported that the phase was promising and presented lower pressure in the column with good selective capacity.

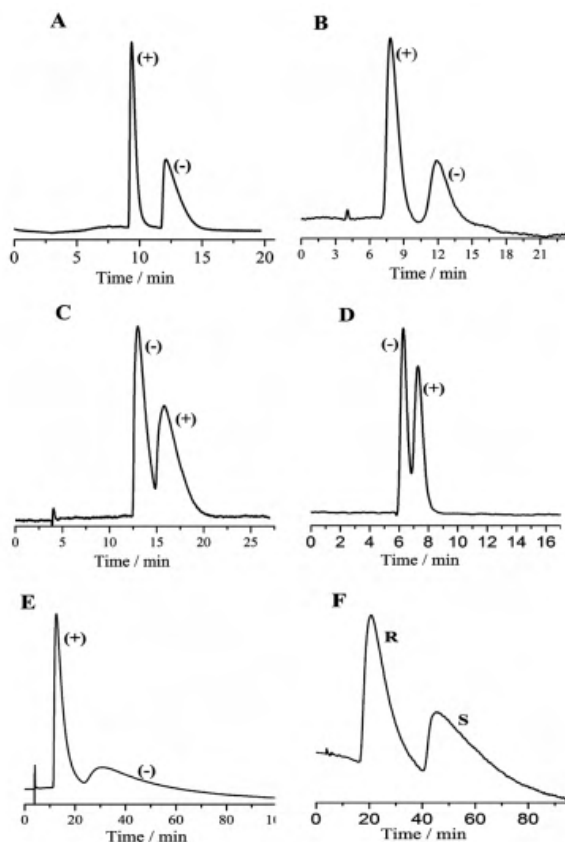


Figure 10. Chromatograms for the separation of racemates: (A) 1-(1-Naphthyl) ethanol, (B) metoprolol, (C) Troger base, (D) trans-tolylene oxide, (E) 1,1'-2-naphthol and (F) Furion. Reprinted (adapted) with permission from (ZHANG et al., 2014). Copyright American Chemical Society.

Hailili et al. (2015) drew attention to a pharmaceutical problem, the chiral recognition to obtain optically pure enantiomers since each enantiomer has physicochemical properties different from its other pair, that is, they have different pharmacological effects and may present toxicity. Faced with this problem, they performed a study for the separation of racemates such as ( $\pm$ )-ibuprofen and ( $\pm$ )-1-phenyl-1,2-ethanediol. They developed a new homochiral structure based on cluster Mn<sub>4</sub>O with ptO and a D-camorphic enantiopure acid. As results, figure 11, it can be seen that the column has excellent chiral recognition ability, peak broadening and retention time can be justified because of the intense interactions of the analytes with the chiral MOF.

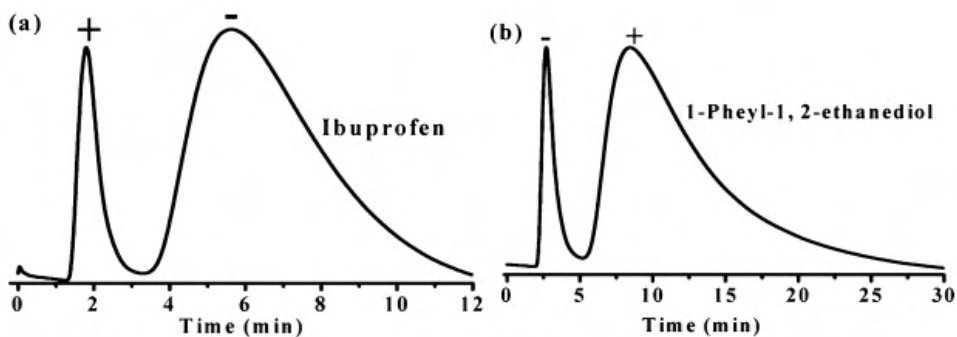


Figure 11. Chromatogram of enantioseparation (a) ( $\pm$ )-ibuprofen and (b) ( $\pm$ )-1-phenyl-1,2-ethanediol. Reprinted (adapted) with permission from (HAILILI *et al.* 2015). Copyright American Chemical Society).

### 2.3 Other Considerations

The composition of the mobile phase has a significant influence on retention time, peak shape, and resolution. In addition to the article by EHRLING *et al.*, 2018, the reviews of VOORDE *et al.*, 2014 and ZANG *et al.*, 2017, conduct studies that discuss the influence of the mobile phase and report on the enantiomeric separations.

## 3 | CONCLUSION

The advances that the MOFs have undergone in the last 10 years are evident, important and essential in the area of adsorption. Because they present unique characteristics, such as: high porosity, selectivity and multiple active sites. MOFs emerged as promising stationary phases for separation on liquid chromatography.

There is an absolute growth in the area of pre-treatment and modification of MOFs that aims to improve their efficiency and activity. The influence on how it will be adsorbed and on what will be adsorbed, after all, the type of treatment and / or modification will define next to the type and composition of the MOF, the active surface area, the dimensions, pore

diffusivity, affinity, high stability and high selectivity.

It is evident the importance and necessity of the advances in the stationary phases with wide selectivity to overcome the problem of non-uniformity of the particles that generate high pressure in the column and lose efficiency.

In summary, further studies are still needed on the subject, however, the observed results show the high potential of the MOFs for application in adsorption and chromatography.

## REFERENCES

Ahmad, R., Wong-Foy, G. A., Matzger, A. J. (2009). Microporous Coordination **Polymers as Selective Sorbents for Liquid Chromatography**, *Langmuir Letter*, 25, 11977-11979.

Alaerts, L., Maes, M., Jacobs, P. A., Denayer, J. F. M., De Vos, D. E. (2008). **Activation of the metal-organic framework MIL-47 for selective adsorption of xylenes and other difunctionalized aromatics**, *Phys. Chem. Chem. Phys.*, 10, 2979-2985.

Ameloot, R., Liekens, A., Alaerts, L., Maes, M., Galarneau, A., Coq, B., Desmet, G., Sels, B. F., Denayer, J. F. M., De Vos, D. E. (2010). **Silica-MOF Composites as a Stationary Phase in Liquid Chromatography**, *Eur. J. Inorg. Chem.*, 2010, 3735-3738.

Chen, S., Li, X. X., Shu, L., Somsundaran, P., Li, J. R. (2017). **The high efficient separation of divinylbenzene and ethylvinylbenzene isomers using high performance liquid chromatography with Fe-based MILs packed columns**, *Chromatogr. A.*, 1510, 25-32.

Collins, C.H., Braga, G.L., Bonato, P.S. (2006). *Fundamentos de Cromatografia*, Editora da UNICAMP, Campinas-SP.

Cychosz, K. A., Wong-Foy, A. G., Matzger, A. J. J. (2008). **Liquid Phase Adsorption by Microporous Coordination Polymers: Removal of Organosulfur Compounds**, *Am. Chem. Soc.*, 130, 6938-6939.

Ehrling, S., Kutzscher, C., Freund, P., Muller, P., Senkovska, I., Kaskel, S. (2018). **MOF@SiO<sub>2</sub> core-shell composites as stationary phase in high performance liquid chromatography**, *Microporous and Mesoporous Materials*, 263, 268-274.

Fu, Y.Y., Yang, C.X., Yan, X.P. (2013). **Fabrication of ZIF-8@SiO<sub>2</sub> Core-Shell Microspheres as the Stationary Phase for High-Performance Liquid Chromatography**, *Eur. J. Inorg. Chem.* 19, 3735-3738.

Hailili, R., Wang, L., Qv, J., Yao, R., Zhang, X.M., Liu, H. (2015). **Planar Mn<sub>4</sub>O cluster homochiral metal-organic framework for HPLC separation of pharmaceutically important ( $\pm$ )-ibuprofen racemate**, *Inorg. Chem. - ACS publication*, 54, 3713-3715.

Hayes, R., Ahmed, A., Edge, T., Zhang, H. (2014). **Core shell particles: Preparation, fundamentals and applications in high performance liquid chromatography**. *Journal of Chromatography A*, 1357, 3652.

Li, H., Eddaoudi, M., O'Keeffe, M., Yaghi, O. M. (1999). **Design and synthesis of an exceptionally stable and highly porous metal-organic framework**, *Nature*, 402, 276- 279.

Li, J. R., Kuppler, R. J., Zhou, H.C. (2009). **Selective gas adsorption and separation in metal-organic frameworks**, *Chem. Soc. Reviews*, 38, 1477–1504.

Lv, Z., Sun, Z., Song, C., Lu, S., Chen, G., You, J. (2016). **Sensitive and background-free determination of thiols from wastewater samples by MOF-5 extraction coupled with high performance liquid chromatography with fluorescence detection using a novel fluorescence probe of carbazole-9-ethyl-2-maleimide**, *Talanta*, 161, 228-237.

Long, J. and Yagui, O. M. (2009). **The pervasive chemistry of metal–organic frameworks**, *Chem. Soc. Reviews*, 38, 1213-1214.

Mukherjee, S., Desai, A. V., Ghosh, S. K. (2018). **Potential of metal–organic frameworks for adsorptive separation of industrially and environmentally relevant liquid mixtures**, *Coordination Chemistry*, 367, 82-126.

Nordin, A. N. H., Racha, S.M., Matsuura, T., Misdan, N., Aimie, N., Sani, A., Ismail, A.F., Mustafa, A. (2015). **Facile modification of ZIF-8 mixed matrix membrane for CO<sub>2</sub>/CH<sub>4</sub> separation: synthesis and preparation**, *RSC Advances*, 5, 43110–43120.

Qin, W., Silvestre, M. E., Brenner-Weiss, G., Wang, Z., Schmitt, S., Hübner, J., Franzreb, M. (2015). **Insights into the Separation Performance of MOFs by High- Performance Liquid Chromatography and In-depth Modelling**, *Sep. and Puri. Technology*, 156, 249-258.

Qu, Q., Xuan, H., Zhang, K., Chen, X., Ding, Y., Feng, S., Xu, Q. J. (2017). **Core-shell silica particles with dendritic pore channels impregnated with zeolite imidazolate framework-8 for high performance liquid chromatography separation**, *Chromatography A.*, 1505, 63-68.

Rocío-Bautista, P., Pino, V., Pasán, J., López-Hernández, I., Ayala, J. H., Ruiz- Pérez, C., Afonso, A. M. (2018). **Insights in the analytical performance of neat metalorganic frameworks in the determination of pollutants of different nature from waters using dispersive miniaturized solid-phase extraction and liquid chromatography**, *Talanta*, 179, 775-783.

Tanaka, K., Muraoka, T., Hirayama, D., Ohnishi, A. (2012). **Highly efficient chromatographic resolution of sulfoxides using a new homochiral MOF–silica composite**, *Chem. Commun.*, 48, 8577- 8579.

Voorde, B. V., Bueken, B., Denayer, J., Vos, D. (2014). **Adsorptive separation on metal–organic frameworks in the liquid phase**, *Chem. Soc. Rev.*, 43, 5766–5788.

Yan, Z., Zhang, W., Gao, J., Lin, Y., Li, J., Lin, Z., Zhang, L. (2015). **Reverse-phase high performance liquid chromatography separation of positional isomers on a MIL-53(Fe) packed column**, *RSC Adv.*, 5, 40094– 40102.

Zhang, M., Xue, X.D., Zhang, J.H., Xie, S.M., Zhang, Y., Yuan, E L. M. (2014). **Nantioselective chromatographic resolution using a homochiral metal-organic framework in HPLC**, *Anal. Methods*, 6, 341–346.

Zhang, Q., Cui, Y., Qian, G., (2017). **Goal directed design of metal-organic frameworks for liquid phase adsorption and separation**, *Coordination Chemistry Reviews*.

Zhang, J., Chen, Z. (2017). **Metal-organic frameworks as stationary phase for application in chromatographic separation**, *Journal of Chromatography*, 1530, 1-18.

Zhu, Y., Ciston, J., Zheng, B., Miao, X., Czarnik, C., Pan, Y., Sougrat, R.(2017). **Unravelling surface and interfacial structures of a metal – organic framework by transmission electron microscopy**, *Nature Materials*, 5, 532-536.



## ÍNDICE REMISSIVO

### A

Adsorção 84, 85, 86, 93, 94, 95, 98, 99, 151  
Adsorvente 84, 94, 95  
Agência de Proteção Ambiental Americana - USEPA 15  
Agência Nacional de Mineração - ANM 145, 153  
Aguas residuales 25  
Águas superficiais 98  
Ambiente aquático 98  
Análise qualitativa 50  
Análise quantitativa 1  
Antioxidante 50, 51, 52, 53, 54, 55, 56, 57  
Argilominerais sintéticos 84

### B

Biocombustíveis 108, 116  
Biomassa 81, 107, 108, 109, 110, 112, 113, 114, 118

### C

Câncer 15, 54  
Catalisador 42, 99, 101, 107, 108, 109, 110, 111, 114, 115, 116, 117, 118, 120, 121, 122, 123, 124, 125, 126, 127  
Combustíveis fósseis 120, 121  
Compostos tóxicos 98  
Comprimido 1, 2, 3, 5, 7, 8, 9, 10, 11  
Contaminantes ambientais 13  
Corantes 17, 97, 98  
Corrosion 129, 130, 131, 134, 135, 136, 137, 138, 139, 140, 141, 142, 143  
Cromatografia gasosa 41, 42, 46, 47, 112, 116

### D

Degradação térmica 109  
Desenvolvimento sustentável 144  
Destilação 40, 41  
Doseamento 1, 6

## **E**

Ecosistemas aquáticos 79

Efeito estufa 121

Efluentes industriais 98

Electrochemical Impedance Spectroscopy -EIS 130

Eletrodo 13, 14, 15, 16, 18, 19, 20, 22, 24

Espectrofotometria 25, 26

Espectroscopia no Infravermelho por Transformada de Fourier - FT IR 109

Essential oils 48, 156, 157

Etanol 43, 50, 52, 120, 121, 126, 127, 184

Eutrofização 79

## **F**

Farmacocinéticos 2

Fármacos 12, 98, 106

Fertilizantes 76, 77, 81, 98

Fitoquímica 50, 52, 53, 54, 55

Flora 51, 150

Fósforo 76, 77, 78, 79, 80, 81, 82, 83

Fotocolorímetro 25, 27, 28, 31, 32, 33, 34, 35, 36, 37, 39

## **G**

Green diesel 108, 116

## **H**

Hidroalogenação 40, 42, 43

Hidrocarbonetos Policíclicos Aromáticos - HPAs 13, 14, 15, 23

High Performance Liquid Chromatography - HPLC 60, 66, 69, 73, 74

## **I**

Impacto ambiental 16, 144, 153

## **L**

Lantanídeos 160, 161, 162, 164, 165, 166, 167, 170, 171, 176, 180, 181, 183, 184, 185

Legislação ambiental 98

## **M**

Material particulado em suspensão 145

Medicamentos 1, 2, 3, 5, 7, 8, 9, 10  
Meio ambiente 13, 14, 23, 81, 98, 107, 120, 121, 146, 148, 152, 153  
Metais pesados 84  
Metais traços 98  
Metal Organic Frameworks - MOF's 58  
Métodos eletroanalíticos 14  
Micro-organismos 152  
Mineração 78, 144, 145, 153

## **N**

Nanoparticles - NP 156, 157, 158, 159, 186  
Normas Reguladoras de Mineração - NRM 145, 153

## **P**

Partículas totais em suspensão - PTS 145  
Pirólise 15, 107, 108, 109, 110, 111, 113, 114, 115, 116, 117, 118  
Planejamento fatorial 40, 41, 42, 43, 44, 45, 46, 48, 49  
Plantas medicinais 51, 56, 57  
Poluentes orgânicos 84, 98  
Poluição atmosférica 145  
Potencial carcinogênico 14

## **R**

Reação de Fenton 97, 99, 101  
Recursos hídricos 81, 98

## **S**

Solução tampão 14, 18  
Supressores químicos 144, 145, 146, 153  
Sustentabilidade 76, 82, 107, 149

## **T**

Taninos 50, 52, 53, 54, 56  
Terpeno 40, 42  
Toxicidade 5, 15, 16, 56, 184  
Troca iônica 84, 85, 147

## V

Voltametria 14, 16, 24

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