Revista Ibero-Americana de Ciências Ambientais
Ibero-American Journal of Environmental Sciences

Nov 2023 a Jan 2024 - v.15 - n.1

ISSN: **2179-6858**

This article is also available online at:
www.sustenere.inf.br

Removal of aquatic toxicity by activated carbon produced from industrial sludge

The valorization of industrial sludge through beneficiation has attracted the attention of several researchers. The expectation is to optimize the reuse and recovery of biosolids generated in wastewater treatment plants by converting them into a product with greater added value, followed by efficient reintegration into the wastewater treatment process. Industry is an important segment of any country's economy for generating employment and quality of life, but it is also a major consumer of natural resources, generating greenhouse gases, solid waste, effluents and sludge. The aim of this work was to review the literature to investigate the synthesis of activated carbon (AC) using industrial sludge as a precursor material, its application in the removal of micropollutants and toxicity in real and synthetic effluents. It was observed that the synthesis route of AC plays a more significant role in their obtainment than the nature of the sludge itself, with reactor temperature and time, activation type, and activator impregnation ratio being the most influential variables. These adsorbents proved to be efficient at removing micropollutants, with up to 99% removal of un. pt-co from textile effluents and toxicity, with up to 100% removal of saline artemia mortality. Therefore, the management of industrial sludge for valorization into value-added material proved to be a viable alternative with proven efficiency in the removal of emerging micropollutants and toxicity. However, the literature is still scarce on the production of CA from industrial sludge and more scientific research is needed, especially with regard to the application of these adsorbents for the removal of aquatic toxicity.

Keywords: Adsorbent; Emerging Micropollutants; Tertiary Treatment; Effluents; Industrial Biosolids.

Remoção de toxicidade aquática por carvão ativado produzido de lodo industrial

A valorização de lodos industriais por beneficiamento tem atraído atenção de vários pesquisadores. A expectativa é a reutilização e agregação de valor ao biossólido gerado nas estações de tratamento de efluentes com sua conversão em produto e reintrodução no tratamento de efluentes. A indústria é o segmento importante na economia de qualquer país para a geração de emprego e qualidade de vida, porém grande consumidora de recursos naturais, geradora de gases de efeito estufa, resíduos sólidos, efluentes e lodos. Assim, o objetivo deste trabalho foi a revisão da literatura para a investigação da síntese de carvão ativado (CA) utilizando lodo industrial como material precursor, sua aplicação na remoção de micropoluentes e a toxicidade em efluentes reais e sintéticos. Constatou-se que a rota de síntese dos CA é mais importante para sua obtenção do que a natureza do próprio lodo, sendo a temperatura e tempo de reator, tipo de ativação e razão de impregnação de ativador como as variáveis mais influentes. Estes adsorventes se mostraram eficiente na remoção de micropoluentes, apresentando até 99% de remoção de un. pt-co de efluentes têxteis e de toxicidade com remoção de até 100% de mortalidade de artemia salina. Logo, o gerenciamento de lodo industrial para a valorização em material de valor agregado mostrou-se alternativa viável com eficiência comprovada na remoção de micropoluentes emergentes e toxicidade. Entretendo a literatura ainda é escassa em produção de CA de lodos industriais e requer mais investigação cientifica principalmente no que tange a aplicação desses adsorventes para remoção de toxicidade aquática.

Palavras-chave: Adsorvente; Micropoluentes Emergentes; Tratamento Terciário; Efluentes; Biossólidos Industriais.

Topic: **Engenharia Sanitária**

Reviewed anonymously in the process of blind peer.

Received: **12/12/2023**
Approved: **05/01/2024**

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DOI: 10.6008/CBPC2179-6858.2024.001.0002

Referencing this:
MONTEIRO, K. A.; SILVA, A. A. S.. Removal of aquatic toxicity by activated carbon produced from industrial sludge. **Revista Ibero Americana de Ciências Ambientais**, v.15, n.1, p.9-24, 2024. DOI: http://doi.org/10.6008/CBPC2179-6858.2024.001.0002

INTRODUCTION

The manufacturing sector is the most critical and basic sector of the economy. This segment is responsible for the largest share of carbon emissions among all industries, yet it is the sector with the greatest impact on national growth and on improving people's quality of life. This sector is characterized by batch and continuous production, with high energy and water consumption, and increasing generation of effluents and sludge (ABIDEMI et al., 2018; QIU et al., 2023).

These effluents have a high chemical oxygen demand associated with the presence of solvents, surfactants, dyes, pharmaceuticals, endocrine disruptors, among others, and their metabolites. The interactions between these substances in effluents are not yet well understood, but it is known that they can affect human health and aquatic fauna, since many of these compounds are considered environmentally persistent and bioaccumulative (LIMA et al., 2022).

Due to the environmental risks of these effluents, there is a need to treat the high volumes, and consequently, considerable quantities of sludge are generated with the need for a final destination that avoids landfills. This semi-solid waste is organic in nature and can be managed in accordance with the circular economy, converting it into a value-added product (GEISSDOERFER et al., 2017; ODA et al., 2021).

Given its composition, sludge has potential as a precursor for activated carbon (AC), which is a promising route for disposing of sludge that eliminates secondary pollution from improper disposal and allows it to be reused in the treatment of gaseous and aqueous streams to remove undesirable compounds (XU et al., 2015).

AC are materials with a high carbon content, made up of heteroatoms, which are highly adsorbent and have a high specific surface area, developed porosity and functional surface chemical groups (ZHOU et al., 2021). AC is widely used in the sanitary sector for tertiary sewage treatment to remove compounds that are persistent in secondary treatment, such as preservatives, metals, dyes, antibiotics, endocrine disruptors, among others (DAOUDA et al., 2021; ELIAS et al., 2021; MANDAL et al., 2021). Treatment by AC can considerably reduce chemical oxygen demand (COD) and mitigate the toxic effect of effluents on the aquatic environment into which they are discharged (GUL et al., 2021).

There is no review in the literature that has investigated the use of industrial sludge for the synthesis of activated carbon, its application in the removal of emerging pollutants and the evaluation of the removal of toxicity linked to these pollutants. Therefore, the aim of this work is to investigate, by selecting and analyzing the literature, the use of industrial sludge for the synthesis of activated carbon and its application as an advanced treatment for the removal of emerging pollutants and the toxicity of these pollutants in synthetic and real effluents. The aim is therefore to highlight an eco-frendly route for obtaining value-added products from industrial sludge, presenting it as a viable alternative for managing this waste.

METHODOLOGY

This study was carried out by selecting and analyzing the scientific literature according to the following criteria (Figure 1):

I. Relevant databases: the bibliometric resources used were Science Direct and Scielo to retrieve articles and book chapters;

II. Period of publication: the publications collected were from 1998 to 2023. The majority of references are from the years 2013 to 2023 (69.74 %), the remaining 30.26 % between the period 1991 and 2009 and a total of 76 literatures consulted;

III. Relevant keywords: the main keywords used in various combinations were "industrial sludge", "activated carbon", "aquatic toxicity" and "emerging micropollutants";

IV. Selection of references based on content analysis: after eliminating articles referring to sanitary sludge, any type of waste recovery other than for the production of activated carbon, the remaining articles and book chapters were analyzed in their entirety.

The studies presented in this paper provide an overview of the valorization of industrial sludge, its characterization, application in the removal of emerging pollutants and evaluation of the removal of toxicity from synthetic and real effluents. This enables a broad understanding of the potential of industrial sludge recovery and the viability of its application for the removal of emerging pollutants and aquatic toxicity.

THEORETICAL DISCUSSION

Industrial Sludge

One of the major challenges facing the 21st century is the transition from the current linear economy to the circular economy, understanding solid waste as an opportunity rather than a liability (SILVESTRI et al., 2021). The sludge generated in industrial sewage treatment plants varies in composition according to the industrial sector and production process.

Industrial sludge from textiles, paper, oil fields and refineries, food, cosmetics, among others, is one of the most important hazardous solid wastes with complex components and high moisture content originating from treatment plants. (GAO et al., 2020). Depending on its industrial origin, this waste can contain greater or lesser amounts of pathogenic microorganisms, heavy metals, oils and greases and other toxic and harmful components that are difficult to degrade and easy to form serious secondary pollution (PENG et al., 2022).

The main physicochemical characteristics determined in industrial sludge are COD (chemical oxygen demand), BOD (biochemical oxygen demand), proximate analysis, elemental analysis, nutrients, Al (aluminum), Fe (iron), Mn (maganese), Zn (zinc), Cd (cadmium), As (arsenic) and Hg (mercury) providing a broad overview of the different elements in sludge (Poinen & Bokhoree, 2022). The composition of these elements in industrial sludge varies according to each industrial activity, as shown in Table 1 (Lee et al., 2018).

The treatment and reuse of sludge is a crucial element of a proper management plan. From a sustainability point of view, resource recovery is considered very beneficial for the economy and the environment (KACPRZAK et al., 2017). Depending on the physical-chemical characteristics of the sludge, some limitations are imposed on its management. For example, soil application is not recommended if the sludge has a high metal content, and pyrolysis is unfeasible for sludge with a high inorganic matter content (WANG et al., 2017).

Table 1: Composition of sludge generated in effluent treatment industrial.

Sludge	DM (%)	Parameters							
		OM (%)	pH	P (mg/g)	Ca (mg/g)	Al (mg/g)	Mg (mg/g)	N (mg/g)	Reference
textile	-	63	7,6	41	3,1	-	7,5	70,9	(ZHANG et al., 2021)
Pulp and cellulose	42,8	-	7,2	3,7	-	-	-	15,8	(GLIŃSKA et al., 2019)
petrochemicals	34,5	69	7,5	-	14,3	-	5,2	-	(AHMADI et al., 2013)
metallurgy	36,73	-	8,3	-	79,80	16,62	-	-	(ZENG et al., 2022)
food	17,9	85	6,7	11	0,2	-	0,2	32	(PASCUAL et al., 2018)
baking	-	85,3	8,3	5,72	1,9	4,2	-	8,2	(YADAV et al., 2019)
cosmetics	34,7	86,4	6,69	-	15	16	3	25,4	(OLIVEIRA et al., 2022)

OM = organic matter; DM = dry matter; pH = hydrogen potential; P = (phosphorus), Ca = (calcium), Al = (aluminum); Mg (magnesium); N (nitrogen).

The most common ways of disposing of industrial sludge are still landfill, dumping in the ocean and land application (WANG et al., 2019). Until recently, land application was seen as one of the most viable alternatives for disposing of sludge, since this waste has nutrients that favor its recycling. However, since the 2000s there has been growing concern about the accumulation of heavy metals in the soil and their leaching into groundwater, leading to a decline in this technique (SAMOLADA et al., 2014).

Due to these environmental liabilities and the complexity of sludge treatment, other reuse solutions are being studied in order to add value to this waste, such as the production of building blocks, biofertilizers, road paving, adsorbent material, among others (DEVI et al., 2017; PACHECO et al., 2012; LIU et al., 2022). Figure 2 shows the characteristics of sludge from different sources that favor adsorbent formation.

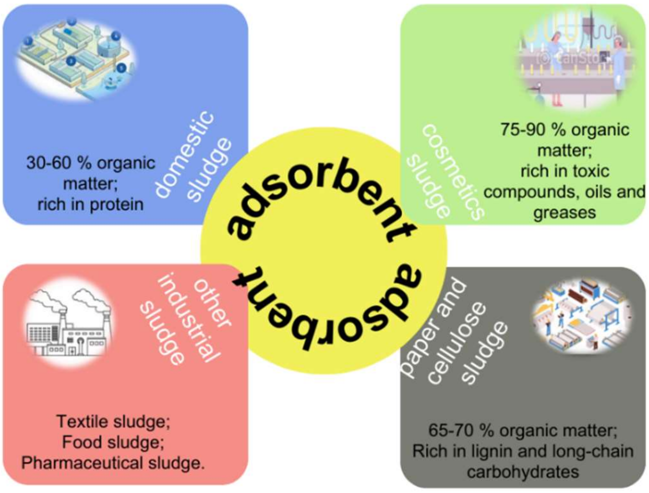

Figure 2: Characteristics of sludge from different sources.

Sludge can be transformed into activated carbon through pyrolysis, and a higher yield is obtained with sludge with a reduced inorganic fraction. During pyrolysis, the heavy metals in the sludge are converted into oxidizable fractions, reducing their bioavailability, ecotoxicity and environmental risk. (JIN et al., 2016; WU et al., 2022).

The AC produced by pyrolysis has a high surface area and large pore volume and has therefore been widely used as an efficient adsorbent for various compounds. In addition, this adsorbent has the advantage of being a value-added material, contributing effectively to sludge management and thus mitigating the environmental impacts of its irregular disposal (SHACKLEY et al., 2011).

Table 2 shows some studies on the synthesis of adsorbent material via pyrolysis of industrial sludge and its efficiency in removing the pollutant of interest. In these studies, the Langmuir and Freundlich isotherms were adjusted to analyze the experimental data and explain the removal mechanism (SANTOS et al., 2022). Based on the studies cited in Table 2, it can be seen that the use of activated carbon obtained from the pyrolysis of industrial sludge is a promising alternative for the removal of various pollutants. With regard to the use of cosmetic sludge, it is rich in carbon and has a significant percentage of organic matter compared to other industrial sludges, making it possible to achieve a high yield in adsorbent synthesis, depending on the synthesis route chosen (MONSALVO et al., 2012; RIBEIRO et al., 2021).

Table 2: Studies with industrial sludge as a precursor material for the synthesis of adsorbents for pollutant removal.

Industrial sludge	Pollutant	%ADS	Isotherm model	Reference
Industrial laundry	Remanzol brilliant reactive blue	84	Freundlich	SILVA et al., 2016
Cosmetics	4-chlorophenol	85	Langmuir	MONSALVO et al., 2012
Wine processing	Cr (III)	-	Langmuir	LI et al., 2004
Metallurgy	Cr (VI)	99	Langmuir	BHATTACHARYA et al., 2006
Fertilizer industry	Acid blue	-	Langmuir	JAIN et al., 2003
Paper and cellulose	Pb+2	95	-	WAJIMA et al., 2011
Cosmetics	Methylene blue	90	Langmuir	RIBEIRO et al., 2021
Pharmacist	Fluoroquinolone	99	Langmuir	WU et al., 2022
Paper and cellulose	COD from industrial wastewater	43	Freundlich	ODA et al., 2021

ADS = adsorption

Activated carbon synthesis route

Activated carbon is a term often used to identify carbon-based adsorbents, which have a high surface area, large porosity, developed internal structure and functional surface groups (LARGITTE et al., 2016; WANG et al., 2021). AC is defined as a material predominantly made up of carbon, in the order of 85-95 %, and small amounts of different elements linked to the carbonaceous matrix, with a structure similar to graphite, being a solid that has well-developed internal porosity comparable to a network of branches, which bifurcate into channels, commonly found in powder or granular form. (MOHAN et al., 2006; RAHAMAN et al., 2007).

This adsorbent is obtained by heat-treating the precursor material and activating it before or after the carbonization process, depending on the synthesis route. Activation can be physical or chemical, thus giving the adsorbent different physical, chemical and textural characteristics. Figure 3 shows a schematic model of the most common chemical and physical activation synthesis routes described in the literature.

The main stages of pre-treatment consist of crushing, sieving and drying, in order to prepare the material in the desired particle size for the synthesis of the adsorbent and to remove water so as not to interfere with activation (BANDOSZ, 2006).

As shown in Table 3, the drying, crushing and sieving operations are used in most of the works found in the literature. Ramya et al. (2019) synthesized activated carbon from industrial tannery sludge and observed that the biomass grinding process and the carbonization temperature were the most influential factors in the formation of the adsorbent's mesoporosity, since the more granular the biomass, the greater the contact surface it will have to react with the activator, enabling greater pore development.

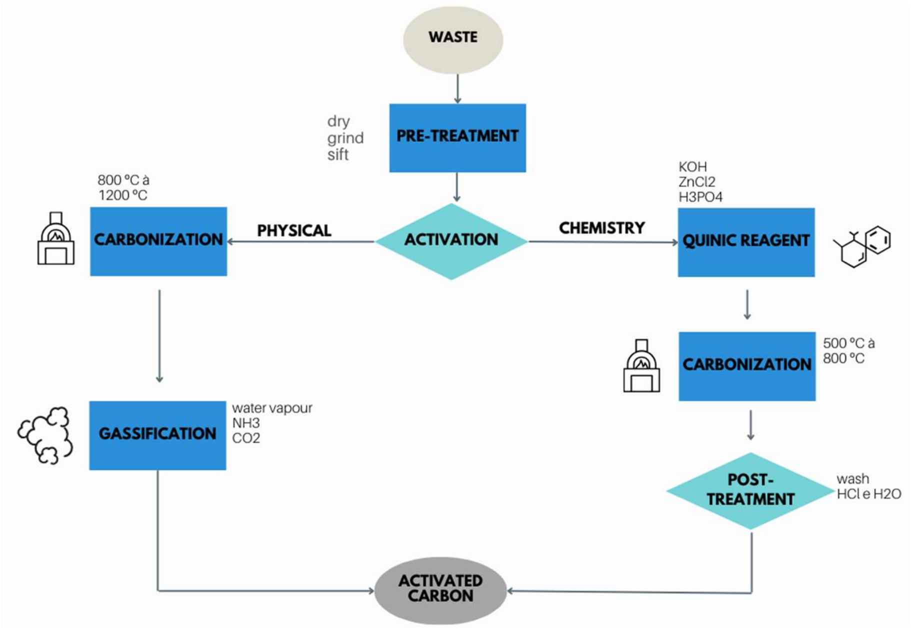

Figure 3: Adsorbent synthesis route by physical and chemical activation.

The activation stage aims to add chemical groups to the surface of the adsorbent and develop pores. This activation can be chemical or physical. In chemical activation, the precursor material is impregnated with an activating agent and then the material is carbonized. Chemical activation favors the creation of pores and sometimes their enlargement, increasing the internal surface area and pore volume through dehydrogenation and degradation of the raw material. (AGULLÓ et al., 2004). Physical activation consists of reacting the already carbonized material with gases such as carbon dioxide (CO_2), steam (H_2O), atmospheric air or a combination of these, mainly with the aim of improving the physical structure of the adsorbent, including factors such as specific surface area, pore size, pore volume, internal micropore volume and surface micropore distribution. (LIU et al., 2022).

The main advantages of chemical activation are lower carbonization temperatures, shorter carbonization times and the development of a porous structure. On the other hand, physical activation does not generate secondary effluents, there is no need for additional expenses related to the purchase of reagents, the process is less corrosive and generates larger surface areas (TENG et al., 1998; YORGUN et al., 2009).

For Ramya et al. (2019) and Santos et al. (2022) the physical activation process with CO_2 favored an increase in the surface area and pore opening of the adsorbent, due to the gasification process which caused the removal of the carbon atoms present inside the particles, as well as the reactions of the CO_2 gas with the carbon, the porosity of the adsorbent material increased due to the evaporation of volatiles during pyrolysis.

On the other hand, Oke et al. (2022) studied the use of phosphoric acid as an activating agent in textile industrial sludge for adsorbent synthesis. The authors found that the activating agent directly influenced the surface area, pore volume and surface chemical groups. After the activation process, the adsorbent had twice the surface area and pore volume of the biomass and this was associated with the action of the acid which acted as a catalyst in breaking the OH bonds formed by the phosphoric esters on the carbon surfaces of the sludge during heat treatment. Wong et al. (2018) studied the synthesis of adsorbent with textile sludge, and inferred that, in addition to the activating agent favoring the development of internal porosity through the formation of stable complexes during activation, the chemical reagent delays the formation of tar and promotes the introduction of oxygenated functionalities to the adsorbent.

Nunthaprechachan et al. (2013) investigated the effect of the type of activating agent ($ZnCl_2$, HNO_3 and KOH), ratio between activating agent and sludge (0.5:1 - 6:1), carbonization temperature (400 - 800 °C) and residence time (0.5 - 2 hours) on the physicochemical properties and adsorption capacity of activated carbon derived from textile sludge. According to the results obtained by the authors, the type of activating agent and the proportion had no significant effect on the constitution (volatiles, ash and fixed carbon) and amount of mesopores of the activated carbons. On the other hand, the parameters analyzed influenced the micropore content and the charcoal activated with KOH had more micropores than that activated with $ZnCl_2$ and HNO_3 , which resulted in a higher BET surface area (S_{BET}).

The same authors also investigated the effect of carbonization on the activation stage. In other words, the authors evaluated two activation conditions: only impregnating the charcoal with the solution containing the activating agent and impregnating the charcoal with the activating agent and carbonizing it again. The results showed that activation with KOH combined with post-impregnation carbonization was the condition that produced activated carbon with the highest surface area and highest dibenzothiophene removal capacity (NUNTHAPRECHACHAN et al., 2013).

As obtained by Nunthaprechachan et al. (2013) activation with $ZnCl_2$ triggered the formation of the oxygenated carbonyl and hydroxyl functional groups of phenols, regardless of the ratio used by the authors. Activation with HNO_3 mainly triggered phenol hydroxyl groups and a small amount of carbonyl, carboxyl and lactone groups, also regardless of the ratio used by the authors. In turn, activation with KOH followed by carbonization generated a greater quantity of oxygenated groups as the ratio between activator and precursor increased, and the main oxygenated group observed for this activator was carbonyl.

The ratio of the activating agent is considered the factor with the greatest impact on the characteristics of the adsorbent during the activation process. Oda et al. (2021) investigated the influence of the activator ratio on the characteristics of the adsorbents produced and found that as the proportion of activator was increased in relation to the amount of sludge, the surface area and pore volume increased and this was associated with the complexation and polymerization reactions of the activator with the sludge, which during the heat treatment process favoured the development of the area and formation of the pores.

Another factor that must be observed when determining the activator ratio is the leachate resulting from the post-treatment process. Santos et al. (2022) in a study of the synthesis of AC from pharmaceutical sludge by physical activation followed by chemical activation using $ZnCl_2$ as the activator, found that 17.7 % of the mass of the adsorbent produced was zinc ions that were leached out during post-treatment, thus identifying a hyperactivation that caused the destruction of the pore walls, resulting in lower porosity values. Therefore, the ideal activator concentration depends directly on the type of sludge and the characteristics of the chemical reagent, and hyperactivation and increased synthesis costs should be avoided (XU et al., 2015).

The pyrolysis stage is the process of heating and decomposing the raw material under an inert atmosphere, through which the sludge can be quickly transformed into natural gas, oil and biochar. This technology is considered one of the most promising for sludge treatment, since the heavy metals present in its constitution are transformed into oxidizable and stable residual fractions, thus reducing the risk of environmental contamination (LIU et al., 2022). The main pyrolysis parameters that directly affect the characteristics of the adsorbent material are pyrolysis temperature, residence time and heating rate (LIU et al., 2022).

Pyrolysis temperature plays an important role in altering the characteristics of adsorbents produced from industrial sludge. Increasing the pyrolysis temperature above the ideal for the waste studied causes an increase in ash generation and a reduction in the yield of the adsorbent, which can be associated with the devolatilization of solid hydrocarbons and the gasification of carbonaceous waste at high temperatures (ZHANG et al., 2011).

Wu et al. (2022) studied the different synthesis parameters that influence the synthesis of adsorbent from pharmaceutical industry sludge and observed that temperature directly influenced the yield and porosity of the activated carbon. The authors found that temperatures above or equal to 500 ºC are necessary to obtain higher yields and mesoporosity, due to the complete volatilization of organic materials. On the other hand, at temperatures above 800 °C, the porous structures formed collapse, probably due to the melting of the pore walls, thus collapsing the pore network formed, in addition to the reduction in yield associated with increased ash generation. The same authors also add that activation at temperatures between 500 °C and 800 °C reduces the C/N (carbon/nitrogen) ratio, which favors the hydrophilicity of the adsorbent, which is beneficial for its application in wastewater treatment.

In agreement with Wu et al. (2022), Orlandi et al. (2017) studied the synthesis of adsorbent from pulp industry sludge by chemical activation with phosphoric acid and investigated the influence of temperature on the yield and chemical characteristics of the adsorbent. The authors inferred that by increasing the temperature from 500 °C to 700 °C, the yield was reduced by 15 %, a result that was already expected, since at high temperatures the amount of organic matter released by the sludge is greater. On the other hand, the concentration of phosphorus compounds was favored as the temperature increased, since at higher temperatures phosphoric acid and its derivatives (polyphosphoric acid) combined with organic species present in the sludge to form polyphosphate esters.

On the other hand, Kante et al. (2008) in an investigation into the influence of temperature on the production of adsorbent activated with waste oil from the sludge of an electroplating industry, found that at a temperature of 950 °C the adsorbents showed higher surface area and pore volume values, which can be associated with the formation of new pores, increased carbon deposition, decomposition of inorganic matter and the release of gases and water that had to find a way out of the solid. It was also possible to state that the increase in the degree of carbonization of the carbonaceous phase and solid state reactions were more likely to occur at 950 °C than at 650 °C. The authors also linked the results to the nature of the industrial sludge used, since at lower temperatures the organic matter decomposition reactions were incomplete.

The length of time spent in the pyrolysis process directly influences the decomposition reactions of the organic matter in the sludge, the development of pores and the costs of the process. In the studies by Wu et al. (2022) on the production of adsorbents from pharmaceutical sludge, the authors found that excessive residence times of over 90 min were not conducive to the development of porosity and surface area, as well as resulting in a reduction in yield and an increase in synthesis costs.

Table 3: Production parameters and characterization of activated carbon derived from industrial sludge in studies found in the literature.

SD	RT	PT.	Pyrolysis				Activation							PO.	Adsorbent characteristics				Reference
			T	TR	HR	FG	AT	AA	T	ASR	FG	TR	HR		SBET	Vt	NI	pHpcz	
STA	Mufla oven	sun-dried and crushed	500	3	-	-	F	CO_2	700	-	0,1	1	-	-	187,21	0,152	-	7,8	(RAMYA et al., 2019)
SF	Quartz vertical reactor	kiln-dried and crushed	800	2	10	100	Q/F	$ZNCl_2$ / CO_2	800	01:01	100	2	10	HCl	458	0,26	-	2	(SANTOS et al., 2022)
SB	Quartz reactor	kiln-dried and crushed	800	1	10	250	Q	$ZnCl_2$	105	01:01	-	24	-	HCl	631,8	0,5098	-	-	(STREIT et al., 2019)
SP	TuBular oven	oven-dried and sieved	800	1,5	10	100	Q	KOH	105	01:01	-	24	-	HCl	697,31	0,631	855	<6	(WU et al., 2022)
SC	Mufla oven	dried and crushed	700	1	3,5	-	Q	NaOH	60	01:01	-	1	-	HCL	74,85	0,07	200	9,6	(ODA et al., 2021)
					3,5					02:01					110,7	0,03	405	9,7	
					15					01:01					50,21	0,05	178	8,5	
					15					02:01					71,59	0,08	189	9,7	
SE	Horizontal oven	Dried and crushed	650	0,5	10	-	Q	Waste oil	-	05:01	-	-	-	-	80	0,15	-	-	(KANTE et al., 2008)
				1											80	0,15			
				2											74	0,139			
			950	0,5											120	0,212			
				1											127	0,212			
				2											124	0,201			
ST	Mufla oven	kiln-dried and crushed	500	1	5	150	Q	H_3PO_4	-	10:01	-	12	-	distilled water	123,65	0,36	-	4,3	(OKE et al., 2022)
SC	Mufla oven	kiln-dried and crushed	500 600 700	1	5	-	Q	H3PO4	85	05:01	-	1	-	$NaHCO_3$ and distilled water	-	-	-	-	(ORLANDI et al., 2017)
ST	Mufla oven	kiln-dried and crushed	650	0,5	5	-	Q	H_2SO_4	30	01:01	-	48	-	HCl and distilled water	221,52	0,1832	-	-	(WONG et al., 2018)
ST	stainless steel fixed bed reactor	oven-dried and sieved	400	2	10	50	Q	KOH	Amb.	0,5:01 - 06:01	-	24	-	distilled water	9,14	0,025	293	-	(NUNTHAPRECHACHAN et al., 2013)
								$ZnCl_2$		0,5:01 - 06:02	-	24	-		5,77	0,016	237	-	
								HNO_3		0,5:01 - 06:03	-	24	-		10,1	0,031	34	-	
								KOH	400 - 600	0,5:01 - 06:04	20	24	10	3M HCl, distilled water	9,14	0,484	1274	-	
								$ZnCl_2$		0,5:01 - 06:05	20	24	10		172	0,191	178	-	
								HNO_3		0,5:01 - 06:06	20	24	10		9,26	0,025	0	-	

T = Temperature (ºC); TR = Residence time (min); ST = Textile sludge; STA = Tannery sludge; SP = Pharmaceutical sludge; SB = Beverage sludge; SC = Cellulose sludge; SA = Alcohol sludge; SE = Electroplating sludge; SBET = BET surface area (m^2/g); Vt = Total pore volume (cm^3/g); NI = Iodine number (mgI_2/g); Q = Chemistry; F = Physics; FTIR = Fourier transform infrared spectroscopy; Amb = environment; SD = sludge; RT= reactor; HR = heating rate (ºC/min); FGN^2 = N^2 gas flow (ml/min); TR = residence time (min); ASR = activant:sludge ratio; AT = activant type; A = Activant; PT = pre-treatment; PO = post-treatment; - = not informed.

Authors such as Méndez et al. (2009) and Yilmaz et al. (2011) have stated that higher pyrolysis temperatures involve shorter residence times. However, in the studies presented in Table 3, this rule is questionable. Kante et al. (2008) and Santos et al. (2022) used high temperatures and long residence times (> 70 min) to obtain materials with a high surface area, which suggests that the need for a longer residence time is directly associated with the nature of the raw material used in the synthesis.

The heating rate dictates the speed at which the reactor will reach pyrolysis temperature and the dehydration and polymerization of the precursor material (MOHAMAD NOR et al., 2013). As shown in Table 3, heating rates reported in the literature for industrial sludge adsorbent synthesis range from 3.5 to 15 °C/min and it can be seen that at lower rates ≤ 10 °C/min the characteristics of the adsorbent are favored.

Oda et al. (2021) evaluated the synthesis of activated carbon from pulp industry sludge and found that the heating rate directly influenced the surface area, and values in the order of 15 ºC/min resulted in significant losses of surface area, pore volume and surface chemical groups, this behaviour being associated with rapid decomposition of the sludge, low dehydration of the sludge before the carbonization reactions and losses in the stabilization of the polymeric compounds.

The post-treatment stage, which includes acid, alkaline and/or distilled water washing, tends to neutralize the surface and reduce the presence of ash in the adsorbent, improving the surface area and porosity values (XU et al., 2015). Table 3 shows that HCl is the main washing agent used in post-treatment. The popularization of this acid is due to the complete dissolution of the oxides (AlO_{23} , FeO_{23} , CaO, etc.) dissolved by washing, leading to the unclogging of pores (ZOU et al., 2013). Santos et al. (2022), Streit et al. (2019) and Wu et al. (2022) related the high surface area values to the post-treatment process with HCl, which promoted the removal of inorganic material, tar and residual activating agent. Santos et al. (2022) and Wu et al. (2022) also found that the acidification of the surface of the adsorbents (pHPCZ < 6) was directly associated with the addition of H+ protons by acid washing.

In view of this, it is important to note that industrial sludge is promising for the production of activated carbon, but because it has different characteristics depending on its industrial origin, more studies are needed to elucidate the phenomena that occur during the synthesis process.

Application of activated carbon to remove toxicity

Toxicology can be understood as the science that seeks to understand the adverse effects caused by chemical substances, especially those of anthropogenic origin, on the organisms present in the ecosystem. (AZEVEDO et al., 2003). The main objectives of this science are to identify the risks associated with substances and to determine under what conditions of exposure these risks are induced (COSTA et al., 2008).

The use of ecotoxicological tests has intensified in recent decades with a view to environmental monitoring, assessing the level of dangerousness and risks of chemical substances and establishing maximum permissible limits for the discharge of liquid effluents into water bodies. (ZAGATTO et al., 2006). Toxicity tests are exposure tests of indicator organisms to certain chemical compounds, substances, sewage, among others, at different concentrations and for a given time. The effects of exposure to the pollutant are assessed through the characteristics presented by the organisms, such as physiological, morphological or behavioral changes.

Toxicity tests carried out in the laboratory are considered to be simple methodologies as they do not require sophisticated and costly analytical methods and provide essential results to indicate the possible toxic effects of contaminants in the environment (COSTA et al., 2008). Due to the high degree of control over the operating conditions of the tests, such as temperature, water quality and photoperiod, it is possible to establish a cause and effect relationship between contaminants, effluents and chemical compounds of environmental risk.

Toxicity tests can be classified in relation to the effects that the organisms may show during the period of exposure to the test as follows (BORRELY et al., 2004; NASCIMENTO et al., 2002):

> Acute toxicity: these are short-term tests covering only part of the organisms' life cycle, usually 24 to 96 h, and the critical effects analyzed are usually the mortality or immobility of the organisms, among others. The main objectives of the test are to determine the intensity of an agent that produces a harmful effect on the organism or the concentration of the test solution;
>
> Chronic toxicity: these are prolonged tests that can cover the entire life cycle of the organism and assess the toxic effects of chemical substances under conditions in which the organisms are able to survive, but undergo altered biological functions such as reproduction, deformity, growth and development of affected eggs;
>
> Short-term chronic toxicity: these are tests with a shorter duration than the traditional chronic test and cover only the sensitive phases of the organism's life cycle, assessing sublethal parameters.

The results of toxicity tests can be expressed as OEC (observed effect concentration), NOEC (non-observed effect concentration), EC (effective concentration), LC (lethal concentration), IC (inhibition concentration) and TU (toxic units). For acute tests, LC50 and EC 50 are the most commonly used, representing the lethal concentration and the effective concentration for 50 % of the exposed organisms, respectively. The inhibitory concentration (IC) is often used in acute or chronic effect tests (COSTA et al., 2008).

These toxicity tests are carried out on various organisms, such as algae, microcrustaceans, fish, polychaetes, oligochaetes, insect larvae, molluscs, echinoderms and bacteria, with methodologies already standardized by international bodies or institutions (POMPÊO et al., 2022; ZAGATTO et al., 2006)

Among the various organisms already standardized for toxicity tests, algae stand out because they are the dominant primary producers in the food chain in the aquatic environment (RODRIGUES et al., 2003). The use of these organisms is important because any change in the dynamics of their communities can affect the upper trophic levels. In addition, the main advantage of using algae in toxicity tests is their short life cycle, which makes it possible to observe toxic effects over several generations (COSTA et al., 2008). In Brazil, toxicity testing with algae (Chlorophyceae) is standardized by the NBR 12648/2018.

The use of toxicity tests as a complementary tool for evaluating the efficiency of activated carbon is of great importance, since adsorption by these carbonaceous materials promotes significant improvements in sewage parameters, but without guaranteeing the removal of toxic compounds. Toxicity assessment therefore makes this possible (FANG et al., 2023). Table 4 shows the application of the activated carbon adsorption technique from different sources for pollutant removal and post-treatment toxicity assessment.

Couto *et al*. (2020) evaluated the removal of chronic bisphenol A toxicity using two industrial activated carbons and *Ceriodaphnia dubia* as a test organism. The authors found that the treatment was efficient in removing the model molecule, reaching approximately 87 and 90 % removal for coconut and bituminous coal, respectively. In relation to the toxicity of bisphenol A, the authors found that for the total removal of chronic toxicity for an initial concentration of 2 mg/L, which means reaching NOEC = 1.56 µg/L, a dose of 535.5 mg/L of coal would be required for treatment with coconut activated carbon and 159.6 mg/L for bituminous activated carbon. The lower dose required for adequate removal of chronic toxicity observed in bituminous coal is associated with greater surface area and lower amounts of impurities.

Gilpavas et al. (2020) evaluated the toxic effect on *Artemia salina* of raw textile effluent, treated with electrocoagulation (EC), the combination of electrochemical oxidation and electrocoagulation (EC + OE) and the sequencing of the two processes plus adsorption by industrial activated carbon (EC + OC + AC). The first effluent was highly toxic, killing 100% of the organisms in 24 hours, and this can be explained by the presence of a high content of dyes and some of the raw materials used in the textile process. The third effluent (EC + OC) had an increase in toxicity compared to the second (EC), reaching 96 % mortality of the species studied, this increase in toxicity may be due to the formation of organochlorine compounds, as well as secondary residual oxidants that remained in the reaction mixture after 30 min of EC. In the fourth effluent (EC + OC + AC) the treatment was highly efficient at removing toxicity, with 0 % mortality, which was attributed to the addition of AC which adsorbed the compounds during the electrocoagulation stage (GILPAVAS et al., 2020).

Table 4: Application of activated carbon from different sources to remove pollutants and post-treatment toxicity assessment.

Origin of AC	Pollutant	Removal (%)	Concentration (mg/L)	Species	Ecotoxicological effect	Reference
Industrial - coconut shell	Bisphenol A	86,2	2	Ceriodaphnia dubia	NOEC=535.5 mg/L	(COUTO et al., 2020)
Bituminous industrial		89,8			NOEC=159.6 mg/L	
Textile industrial sludge	Real textile effluent	>96	1310 (un. Pt-Co)	Artemia salina	Mortality at 0% after sequential treatment of the three processes.	(GILPAVAS et al., 2020)
		99	1399 (un. Pt-Co)			(GILPAVAS et al., 2019)
Sludge from the dairy industry	Erofloxacin	98	20	Escherichia coli	Increase in bacterial community after treatment with activated charcoal; 34% increase in microbial community	(FANG et al., 2023)

CL$_{50}$ lethal concentration to 50% of the test organisms; NOEC concentration of no observed effect; Pt-Co: platinum cobalt scale; AC = activated carbon.

The same authors investigated the combination of electrocoagulation (EC) with photo-Fenton (PF) and activated carbon (AC) processes for the removal of color from the same industrial effluent and toxicity analysis for the species *Artemia* salina (GILPAVAS et al., 2019). They found that after the coupled treatment of electrocoagulation and photo-Fenton (EC + PF), 99% of the color in the effluent was removed. The degradation of the dye molecule was also noticeable, as the main adsorption bands of the raw effluent were eliminated after the coupled treatment (EC + PF), due to the fact that some of the organic compounds were degraded and/or oxidized by the oxidizing species generated during the photo-Fenton process. With regard to the toxic effect on *Artemia salina of the* four effluents studied, it was found that:

> I) Raw effluent: was highly toxic to the species studied, causing 100 % mortality;
>
> II) Effluent treated by EC: toxicity was not altered in relation to the previous effluent, due to the high COD, and concentration of residual chlorine/hypochlorite and toxic intermediates;
>
> III) Effluent treated by EC+PF: mortality of the species fell to 50 %, i.e. still toxic due to the presence of intermediate toxic by-products as well as secondary residual oxidants that remained in the reaction mixture;
>
> IV) Effluent treated by EC + PF + CA: completely removed the toxicity of the effluent, with 0 % mortality of the species, this result being associated with activated carbon which can react with free chlorine to produce organic compounds containing oxygen on the surface of the carbon or CO_2 as the end product.

Fang et al. (2023) investigated the use of sludge from a dairy industry treatment plant for the synthesis of activated carbon, its use without and as a support for peroxydisulfate for the removal of fluoroquinolones and an evaluation of the toxicity of the treatments using bacteria as a test organism. The authors found that the use of activated carbon without the use of peroxidisulphate was efficient in removing erofloxacin, but the number of bacterial colonies was lower than the treatment with activated carbon with peroxidisulphate.

When activated carbon was used as a catalytic support for peroxidisulfate, the treatment was efficient in degrading erofloxacin molecules up to its eighth by-product, reducing the availability and toxicity of this compound for the organism under study, with a 98 % reduction in erofloxacin molecules. The authors attributed the higher number of bacterial colonies (34 % more) in this treatment compared to activated charcoal alone to the availability of the charcoal pores to adsorb the by-products of the degradation of the model molecule, further reducing its availability to cause toxicity in the aqueous environment (FANG et al., 2023).

CONCLUSIONS

This work has shown that industrial sludge has the characteristics and potential to become a value-added material, with proven applications for removing emerging pollutants. The surface area $S_{BET,}$ the main characteristic of activated carbon obtained from industrial sludge, is directly influenced by the synthesis route, with the main variables being reactor temperature, reactor time, type of activation and activator impregnation ratio.

Activated carbon obtained from industrial sludge proved to be efficient for adsorbing micropollutants, with an efficiency of over 40% and higher efficiencies for removing drugs and heavy metals. Tertiary treatment using activated carbon from industrial sludge proved to be efficient in reducing the concentration of micropollutants and toxicity levels to undetectable levels.

In view of the above, other studies need to be carried out to explore the gaps in the production of activated carbon using industrial sludge and its applications, such as:

> Identification of the optimized conditions for synthesizing adsorbents from different industrial sludges;
>
> Application of the adsorbents in real effluents under dynamic conditions, using reactors, with a variety of polluting molecules, to ascertain their efficiency in real systems and competitive sorption.
>
> Alternatives for disposing of and/or reusing the adsorbent after its life cycle has come to an end, preferably by routes that cause less economic and environmental impact.
>
> Toxicity removal studies using industrial sludge-based adsorbents with toxicological tests varying between different trophic levels.
>
> Economic feasibility studies for the synthesis and application of adsorbent material based on industrial sludge.

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