Chem Rep, 2022, 4(1): 210-217
DOI: 10.25082/CR.2022.01.001

RESEARCH ARTICLE

Evaluation of an adsorption process for the treatment of leachates using biopolymers extracted from organic waste obtained from the poultry industry

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Received: April 9, 2022;
Accepted: May 29, 2022;
Published: June 2, 2022.

Citation: Tarazona Y, Rojas-Valencia MN and Araiza-Aguilar JA. Evaluation of an adsorption process for the treatment of leachates using biopolymers extracted from organic waste obtained from the poultry industry. *Chem Rep*, 2022, **4**(1): 210-217. https://doi.org/10.25082/CR.2022.01.001

Abstract: The adsorption capacity of three eggshell bioadsorbents was evaluated to remove contaminants from raw leachate. Optimal conditions for the removal of suspended solids, color, and organic compounds, as COD, were achieved by batch experiments with three levels of pH and absorbent concentrations. Kinetic studies and isotherms were developed to understand the behavior of COD removal by the bioadsorbents. The chemical and physical characterizations indicate the leachate used in the present study had characteristics between mature and intermediate leachates. The optimal adsorption conditions were pH 2.0 and 1.0 gram (0.5 g/L) of adsorbent. Adsorbent M showed the best adsorption capacities, removing 99.06% (1446 NTU) of turbidity, 86.25% (4140 UPt-Co) of color and 54.56% of COD (1530mg/L). The data obtained through the kinetic and isothermal tests were better fitted to the pseudo first order and Langmuir models, with an equilibrium adsorption capacity (Qe) of 139 mg of COD/g of adsorbent and a specific speed of 1.51 min^{-1}.

Keywords: bioadsorbent, biopolymers, leachates, organic waste

1 Introduction

The goal of the circular economy is to reduce waste and to establish a continuous use of resources, considering waste as a resource for generating value-added products [1]. The study of economic and widely available waste materials as adsorption matrixes for the removal of selected constituents of concern from water and wastewater have been intensified in the last decades [2–4]. The capacity of eggshells as an adsorbent material for removing contaminants from wastewater and synthetic water has been shown through different adsorption process configurations. Metallic ions, such as cadmium, iron, and chromium, among others, along with phosphates, cyanides, and artificial dyes such as methylene blue, brilliant green, and crystal violet are some compounds that have been removed using eggshells as bioadsorbents [5–11].

According to the United Nations Food and Agriculture Organization (FAOSTAT) Statistics Directorate, the world production of chicken eggs was around 73.8 million tons in 2013, of which about 3% corresponded to the Mexican Republic, with a production exceeding 2.2 million tons per year.

Worldwide, is estimated that the eggshells waste production of is around 50,000 tons per year [12]. In Mexico, although official data on the amounts of waste generated by this economic sector in Mexico are not available, given such high production levels, it is estimated that eggshell waste is one of the most abundant waste materials coming from the food industry in Mexico.

Despite its unique properties, valorization strategies of eggshell waste are limited. The best-known valorization scenario is the use of these wastes in composting plants to produce marketable fertilizers and the agro-industrial use of eggshells as a crop enhancer or raw material for prebiotics.

Practices such as landfill disposal are still the primary way the eggshell management, nevertheless, in the absence of proper management practices, eggshells can cause serious environmental problems [13–16].

These eggshell waste management considerations and the need for economic and widely available adsorbent materials for leachate treatment, emphasizing obvious advantages.

First, in contrast with the conventional methods where expensive chemical compounds that can be toxic for aquatic organisms are used, the implementation of biological materials as treatment options in some processes translates into cost reduction, ease of use and absence of materials that are difficult to degrade [17, 18].

This research discloses the use of eggshell waste – a potential source of contamination – to address the environmental issues caused by the current technologies used in leachates treatment and the challenges related to eggshell waste management. It is well known that eggshell residues, if not properly managed, can become sources of toxic compounds and ecological niches for insects, parasites, and pathogenic microorganisms, giving rise to potential ecological and public health problems. [1, 13, 14].

Finally, because these wastes have no economic value in most cases, the cost of the technological implementation of these wastes in the manufacture of active biopolymers used in adsorption contactors would be notably lower compared to the reagents and materials that are conventionally employed for such purpose. This would be especially relevant in countries with undeveloped economies leading to a reduction of the environmental impact caused by these by-products and the improvement of the environment, health, and quality of life of near populations [9, 13, 14].

Therefore, the objective of this work was to evaluate the removal capacity of some contaminants present in leachate from the Bordo Poniente Sanitary Landfill (RSBP), through batch adsorption tests using absorbents synthesized from eggshells.

2 Materials and methods

The methodology was divided into four stages

2.1 Sampling

This study was carried out with leachate samples from the Bordo Poniente Sanitary Landfill (RSBP), located in the Federal Zone of Lake Texcoco, east of Mexico City International Airport, between19° 26’ 09.36” and 19° 29’ 09.22” N and 99°00’ 14.51” and 99° 02’ 36.21” W. The geographical location, distribution of the RSBP stages and identification of the sampling points are shown in Figure 1.

The samplings were carried out during eight months, covering a dry and rainy period, in ditches 1 and 3 of RSBP stage III, in accordance with the guidelines of the Official Mexican Standard for water and wastewater NMX-AA-030/1-SCFI-2012, conditioned to the practice and behavior of leachates. After each sampling, the samples were stored in a cold room at a temperature of approximately 4°C during the study time.

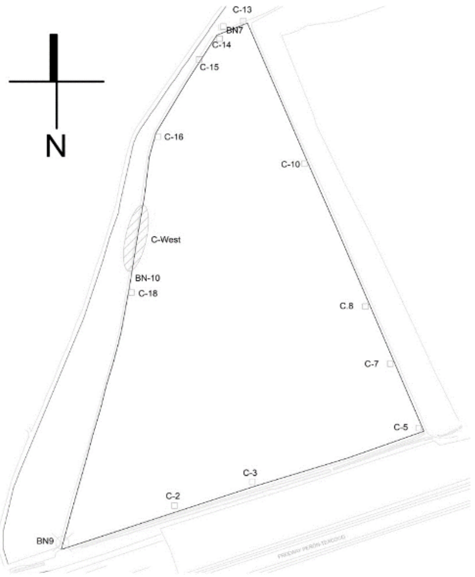

Figure 1 RSBP STAGE III. Location of the sampling points used in the project. Source: Own elaboration from Alcantar, (2015).

2.2 Leachate characterization

The characterization of the samples was carried out by evaluating pH, turbidity, color (465nm), COD, BOD_5, barium, cadmium, chromium, mercury, nickel and lead in crude leachate samples according to the methodologies described in current national regulations for wastewater characterization (Table 1).

Table 1 Regulations used for the characterization of the determined parameters of the leachates

No.	Parameter	Reference Standard
1	pH	NMX-AA-008-SCFI-2011
2	COD	NMX-AA-030-SCFI-2012
3	BOD_5	NMX-AA-028-SCFI-2001
4	Color	NMX-AA.017-1980
5	Turbidity	NMX-AA-038-2001
6	Metals	NMX-AA-051-2001
7	Ammoniacal N	NMX-AA-026-SCFI-2010

2.3 Manufacture of adsorbent biopolymers from eggshell

Three commercial eggshell waste adequacy processes were developed, called bioadsorbents L, M, S, each with different electrical grinding times and sieving processes. L was made with a grinding time of 30 seconds and sieved though a standard No. 40 sieve; M was made with a grinding time of 60 seconds, sieved through a standard No. 60 sieve and S was made with a grinding time of 90 seconds, and sieved through a standard No. 100 sieve. The methodology described by Hormaza and Suarez (2009) [9] and Zhang et al. (2017) [6] was used. Figure 5 describes the process used for obtaining the adsorbents.

2.4 Development and optimization of the purification processes

2.4.1 Adsorption

The adsorption tests were carried out discontinuously, according to the methodology of Correa (2014), using biopolymers made from eggshell residues as bioadsorbent.

2.4.2 Determination of optimal adsorption conditions

The optimal adsorption conditions determined were the pH of the leachate, the amount of bioadsorbent and the contact time using the methodology described in Table 2.

Table 2 Description of the discontinuous test for adsorption processes

No.	Process	Activity
1	Sample placement	200 mL of leachate were poured in an Erlenmeyer flask.
2	pH Modifications	The pH of the leachate was modified with sulfuric acid (37%) until reaching the value to be evaluated.
3	Addition of adsorbents	The masses of adsorbents to be evaluated were added
4	Shaking	
5	Filtration	The samples were filtered through a number 1 filter
6	Sample extraction	150 mL-samples were extracted
7	Conservation	Stored at 4ºC until characterization.

Note: Based on Correa (2014)

Initially, the optimal pH and quantity of adsorbent were determined for the three bioadsorbents (425, 250 and 100 μM) using a contact time of 30 minutes at 1500 rpm. Two leachate pH (2.0 and 4.0) and five masses of the three adsorbents (1.0, 2.5, 5.0, 7.5 and 10 grams) were assessed through tests in triplicate according to the methodology described in Table 2.

2.5 Tests of adsorption kinetics and adsorption isotherms

2.5.1 Adsorption kinetics

The kinetic studies were carried out using the most efficient adsorbent under the optimal conditions determined by carrying out tests in triplicate according to the methodology described in Table 2. Placing 500 mL of sample in a 1 L Erlenmeyer flask, applying constant agitation at 1500 rpm, taking samples during defined time intervals, until observing the equilibrium in the COD concentrations.

2.5.2 Adsorption isotherms

Adsorption isotherms were carried out using the equilibrium conditions determined by kinetic tests. 500 mL of sample were placed in a 1 L Erlenmeyer flask, under constant stirring at 1500 rpm and equilibrium time. The amounts of adsorbent were varied from 1 to 10 grams, in triplicate tests according to the methodology described in Table 2.

2.5.3 Modeling of adsorption kinetics and isotherms

The data obtained in the kinetic tests were fitted to the pseudo-first order, pseudo-second order, Elovich and intraparticle diffusion models and the results obtained in the adsorption isotherms were fitted to the linearized form of the Freundlich and Langmuir models (Table 3).

Table 3 Kinetic and isothermal adsorption models for COD removal in the evaluated leachate samples

Model	Equation
Pseudo-first order	$q_1 = q_2\left(1 - e^{-kji}\right)$
Pseudo-second order	$q_1 = \dfrac{1}{\dfrac{1}{q_c^2{}_{k_2}} + \dfrac{t}{q_c}}$
Elovich	$q_1 = \frac{1}{b}\ln(ab) + \frac{1}{b}\ln t$
Intraparticle diffusion	$q_1 = k_f S^{\frac{1}{s}}$
Freundlich (Linealized form)	$ln\ q_c = ln\ K_c + \frac{1}{b}\ ln\ C_r$
Langmuir (Linealized form)	$\frac{1}{q_c} = \frac{1}{bq_m}\frac{1}{C_e} + \frac{1}{q_m}$

Note: Based on Duran *et al.* (2018) [19]

3 Results

3.1 Leachate characterization

Table 4 shows the results of the characterization of crude leachate samples from the RSBP ditch.

Table 4 characterization and treatments

No.	Parameter	Characterization
1	pH	8.45
2	COD (mg/L)	2800
3	BOD_5 (mg/L)	348
4	Turbidity (UNT)	1640
5	Color (U Pt-Co)	4800
6	Barium (mg/L)	0.46
7	Cadmium (mg/L)	0.00
8	Chromium (mg/L)	0.32
9	Mercury (mg/L)	0.00
10	Nickel (mg/L)	0.32
11	Lead (mg/L)	0.52

RSBP stage III was determined based on the characterization of the samples, the leachate had characteristics between mature leachate (older than 10 years) and intermediate leachate (5-10 years), despite being contained in cells older than 20 years [20].

It had a pH of 8.45, which is within the limits defined by Lozada et al. 2014 for both mature leachate and intermediate leachate. With regard to parameters indicating organic matter, both COD and BOD_5 showed values between those described for mature leachates and intermediate leachates, with BOD_5 values of 348 mg/L and COD values of 2,800 mg/L.

3.2 Manufacture of bioadsorbents from eggshell waste

During the bioadsorbent manufacturing process, three different products were developed. Figure 2 shows the three different bioadsorbents classified according to the differences in particle sizes: between 425 and 300μm for bioadsorbent L, between 300 and 150μm for bioadsorbent M, and smaller than 150μm for bioadsorbent S.


a: Bioadsorbent L b: Bioadsorbent M c: Bioadsorbent S

Figure 2 Bioadsorbents from eggshell waste

3.3 Development and optimization of the purification processes

Optimal adsorption conditions for leachate pH, amount of adsorbent and contact time were determined using the three bioadsorbents.

Figure 3 and 4 shows the residual turbidity in the leachates using the three bioadsorbents at pH 2.0 and 4.0, respectively. At pH 2, adsorbents L, M and S reached significant turbidity removals of 98.81% (bioadsorbent L), 99.06 % (bioadsorbent M) and 98.73 % (bioadsorbent S). At pH 4, removals were 98.24%, 98.2% and 98.8% for bioadsorbents L, M and S, respectively.

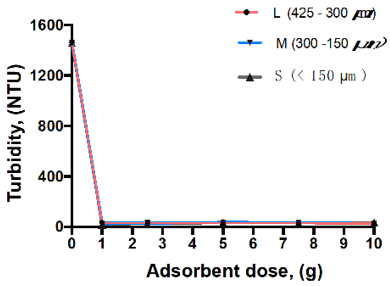

Figure 3 Residual turbidity in optimization tests at pH 2

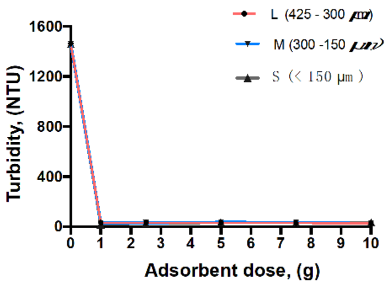

Figure 4 Residual turbidity in optimization tests at pH 4

Figure 5 and 6 show the residual color of the leachates after the adsorption processes using the three bioadsorbents, at pH 2 and 4.

The color removal achieved by the three bioadsorbents was higher at pH 2. Color removal achieved at pH 2 was 85.00% using bioadsorbents L and S and about 86.25% using bioadsorbent M, while color removal achieved at pH 4 was 68.54, 58.95 and 57.70% using adsorbent L, M and S, respectively.

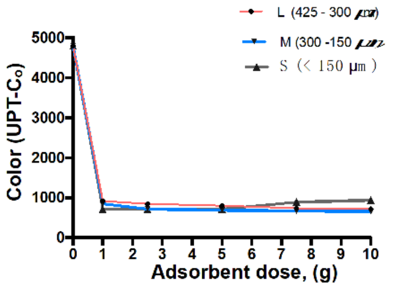

Figure 5 Residual color in optimization tests at pH 2.0

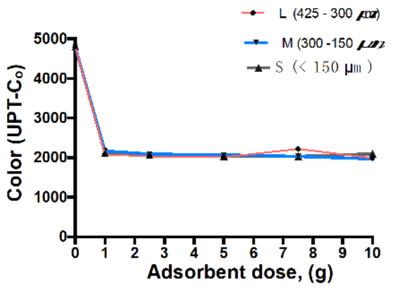

Figure 6 Residual color in optimization tests at pH 4.0

On the other hand, Figure 7 and 8 show residual COD in the adsorption tests at pH 2 and 4. It can be seen that an effect similar to that of color removal occurs, where the pH of the leachate plays an important role in the adsorption process as described by Hormaza and Suarez, (2009) [9] and Novelo et al. (2002), who obtained higher efficiencies in the removal of dyes and COD operating at low pH, using eggshells and activated carbon as adsorbents in the treatment of artificial water and leachate, respectively.

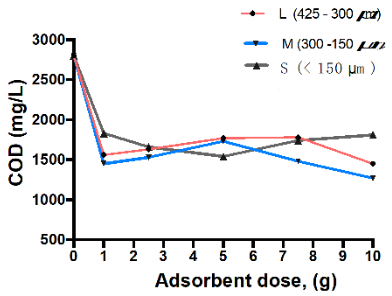

Figure 7 Residual COD in optimization tests at pH 2.0

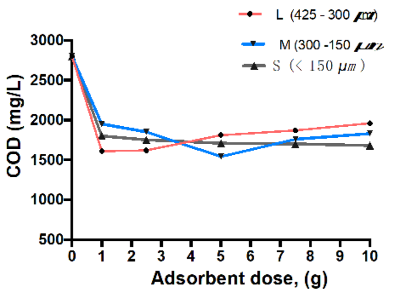

Figure 8 Residual COD in optimization tests at pH 4.0

Based on the optimization tests, it was determined that the optimal conditions were pH 2 and 1 gram of adsorbent (0.5 g/L), reaching maximum removal efficiencies of 99.06% of suspended solids, 86.25% of color and 54.56% of COD, using adsorbent M.

According to Pettinato et al. (2015), the adsorbent potential of the eggshell is attributed to its structural and chemical conformation, which is composed of 98% inorganic compounds such as calcium carbonate ($CaCO_3$) and 2% organic substances, which provide porosity and adsorption capacity to the material. In the same way, it is known that factors such as the pH of the medium, the protonation of active sites and the amount of adsorbent, are factors of transcendental importance in the adsorption process due to their relationship with the proportion of active sites available to carry out the process. Indeed, the variations found in the adsorption capacities of the three bioadsorbents can be associated with these factors.

Baláž et al. (2016) [16] describe that particle size can influence the contact surface area and therefore the availability of active sites. This fact could explain why bioadsorbent M, with particles between 300 and 150μm, showed better efficiencies than bioadsorbents L and S, with different particle sizes.

Compared to the results obtained by other authors with different adsorbent matrices, eggshell residues offer interesting efficiencies.

Charles et al. (2016) who compared, among others, the use of a resin in an ion exchange process and the use of starch crosslinking as an adsorbent matrix in adsorption processes, obtained in both cases lower efficiencies, which did not exceed 40% of COD removal, compared to the efficiencies obtained in the present study.

Some studies have used high-efficiency conventional adsorbents such as activated carbon and reported removal capacities in COD indexes close to 68%, which exceed the efficiencies achieved by eggshell residues in this study by 13%. However, the low cost of eggshell, compared to the high cost of activated carbon, and the ease of application of eggshell waste as an adsorbent, due to the absence of activation processes, offer obvious advantages over the conventional adsorbent.

3.4 Tests of adsorption kinetics and isotherms

3.4.1 Adsorption kinetics

The kinetic data obtained under the optimal conditions of pH and amount of adsorbent using the bioadsorbent M, presented a better fit to the pseudo-first order model, offering irreversible adsorption processes and establishing an adsorption process directly proportional to the contact time, where the amount of adsorbate removed increases as time progresses.

In this sense, Pettinato et al. (2015) state that the adsorption process observed with eggshells is carried out by means of an ion exchange mechanism, where the substances are concentrated on the surface of the adsorbate, because of the influence of electrostatic forces. The fit model as well as the experimental data are shown in Figure 9 and 10.

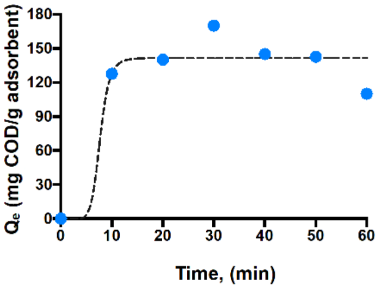

Figure 9 Kinetic adjustment to the pseudo-first order model

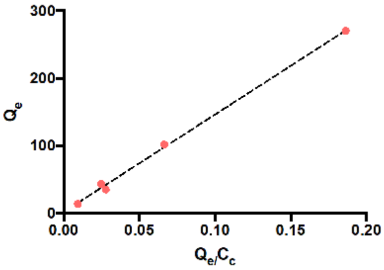

Figure 10 Isotherm fit to the Langmuir model

According to the constants obtained by the pseudo-first order model, the adsorption process will have a specific speed of 1.51 min^{-1} (Equilibrium constant K1) and an equilibrium adsorption capacity (Qe) of 139 mg COD/g of adsorbent, data that graphically coincide with those experimentally obtained. In the same way, it is possible to determine that the contact time necessary to reach the adsorption capacity equilibrium occurs in 20-30 minutes with the bioadsorbent M, under the described adsorption conditions.

3.4.2 Adsorption isotherms

The data obtained using bioadsorbent M, under optimal conditions of pH, amount of adsorbent and a contact time of 20 minutes, presented a better fit to the Langmuir empirical model (see Figure 10) than to the Freundlich model.

These results coincide with those obtained by Hormaza and Suarez (2009) [9], who established in the same way that the data of the eggshell adsorption capacities show a better fit to this model, in which an adsorption process is carried out by equivalent active sites uniformly distributed over the adsorbent material. This allows the establishment of a monolayer adsorption process in which the adsorbate is attracted to the free active sites on the surface of the adsorbent, and once these sites are occupied, the adsorption process terminates, defining the surface of the adsorbent as the limiting factor in the process. The results of the Adsorption Kinetics and Average Adsorption Isotherms of three repetitions are shown in Table 5.

Table 5 Adsorption Kinetics and Adsorption Isotherms, average of three replicates

Test	Turbidity (FNU)	Color (PtCo)	COD	Ag	Al	As	B	Ba	Cd	Cr	Ni	Pb
			(mg/L)									
Adsorption kinetics												
0	23	149	209	0	1.35	0	5.46	0.46	0	0.32	0.35	0.6
1	21.4	118	206	0	1.35	0		0.4	0	0.33	0.34	0.5
2	22	118	193	0	1.34	0		0.32	0	0.31	0.32	0.6
3	23.6	94	221	0	1.34	0		0.27	0	0.3	0.33	0.5
4	22.8	107	195	0	1.34	0		0.12	0	0.27	0.29	0.4
5	19.9	138	210	0	1.33	0	5.04	0.09	0	0.3	0.27	0.45
6	21	98	205	0	1.3	0		0.09	0	0.25	0.27	0.37
7	21.3	110	206	0	1.27	0		0.1	0	0.23	0.25	0.34
Adsorption Isotherms												
1	24	151	211	0	1.49	0	5.45	0.43	0	0.29	0.32	0.65
2	20	129	209	0	1.46	0	5.42	0.14	0	0.23	0.3	0.63
3	21,2	91	197	0	1.44	0	5.41	0.07	0	0.22	0.29	0.58
4	11.1	127	203	0	1.35	0	4.66	0.07	0	0.22	0.29	0.57
5	13.3	161	219	0	1.33	0	4.66	0.01	0	0.21	0.27	0.55
6	13.9	179	181	0	1.28	0	4.35	0.01	0	0.23	0.29	0.49

4 Conclusions

The experiments carried out demonstrate the potential of eggshell residues as bioadsorbents in leachate purification systems.

The pH and adsorbent concentration affect the adsorption capacity of eggshell adsorbents for the removal of suspended solids as turbidity, color, and organic matter as COD. Optimization tests showed that adjustment of leachate pH to 2 and using adsorbent concentrations of 0.5 g/L (1 g) improve egg shell adsorption efficiency.

The size of the eggshell adsorbents impacts its adsorption capacity. Particles between 300 and 150 μm (adsorbent M) showed the best performance in the present study and reached maximum removal efficiencies of 99.06% (1446 NTU) of suspended solids, 86.25% (4140 UPt-Co) color, and 54.56% COD (1530 mg/L).

Experimental kinetic data analysis showed that eggshell adsorbents had a better fit to the pseudo-first order model. Adsorbent M reached an equilibrium adsorption capacity (Qe) of 139 mg COD / g of adsorbent at a specific rate of 1.51 min^{-1}. The data obtained through the adsorption isotherms presented a better fit to the Langmuir model.

Conflict of interest

The authors declare that they have no conflict of interest.

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