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Detection of chemical compounds in water for agricultural use in Xochimilco and Tlahuac

Detección de compuestos químicos en agua de uso agropecuario en Xochimilco y Tláhuac

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Resumen

La producción agropecuaria es la principal fuente de contaminación de los cuerpos de agua debido al uso excesivo de agroquímicos y el inadecuado manejo de los desechos orgánicos de los animales. El agua utilizada para riego agrícola puede contener varios compuestos químicos, como sulfatos [SO_4^{2-}], fosfatos [PO_4^{3-}], fenoles [Ph] y 2-clorofenol [ClPh]. La legislación Mexicana establece límites de [130 y 250], [0.05 y 5], [0.1 y 0.3] y [0.04] ppm, respectivamente. Una combinación de estos, especialmente PO_4^{3-} (0.5 ppm), provoca la mortalidad de ecosistemas acuáticos y terrestres. Las alcaldías de Xochimilco y Tláhuac, en la Ciudad de México, producen hortalizas, entre otros alimentos, utilizando agua del canal con agua residual para su riego. Por ello, es necesario analizar la presencia de estos compuestos, empleando métodos analíticos sensibles para su detección de acuerdo con las Normas Oficiales Mexicanas. El objetivo de este trabajo fue detectar las concentraciones de SO_4^{2-}, PO_4^{3-}, Ph y ClPh presentes en el agua de uso agropecuario de Xochimilco y Tláhuac. Se colectaron un total de 20 muestras de agua de uso agropecuario, provenientes de 20 puntos geográficos de los cuerpos de agua de Xochimilco y Tláhuac. La determinación de SO_4^{2-},

PO_4^{3-} y fenoles totales se realizó por Espectrofotometría UV-VIS, utilizando los métodos: azul de molibdeno, turbidimétrico modificado y el directo espectrofotométrico, respectivamente. El Ph y ClPh se identificaron y cuantificaron por un nuevo método por HPLC. El agua de uso agrícola presentó niveles máximos de SO_4^{2-}, PO_4^{3-}, fenoles totales y ClPh de 277.56 ± 0.3, 4.71 ± 0.004, 1.44 ± 0.01 y 0.965 ± 0.002 ppm, respectivamente. Para el agua de uso pecuario, los valores máximos fueron 461.03 ± 3.58, 1.71 ± 0.05, 0.78 ± 0.005 y 0.119 ± 0.004 ppm, respectivamente. El agua de uso agropecuario de Xochimilco y Tláhuac tiene altas concentraciones de SO_4^{2-}, PO_4^{3-}, Ph y ClPh. En algunos casos, excede los límites establecidos por la regulación Mexicana. Por lo tanto, se considera no apta para estas actividades ya que representa un riesgo para el medio ambiente, la salud animal y la salud humana.

Palabras clave: agua de uso agropecuario, clorofenol, fenol, fosfatos, sulfatos.

Abstract

Agricultural production is the main source of contamination of water bodies due to the excessive use of agrochemicals and inadequate handling of organic waste from animals. Water used for agricultural irrigation can contain various chemical compounds, such as sulfates [SO_4^{2-}], phosphates [PO_4^{3-}], phenols [Ph] and 2-Chlorophenol [ClPh]. Mexican legislation establishes limits of [130 and 250], [0.05 and 5], [0.1 and 0.3], and [0.04] ppm, respectively. A combination of these, especially PO_4^{3-} (0.5 ppm), causes the mortality of aquatic and terrestrial ecosystems. The municipalities of Xochimilco and Tlahuac, in Mexico City, produce vegetables, among other foods, using water from the canal with residual water for irrigation. Therefore, it is necessary to analyze the presence of these compounds, using sensitive analytical methods for their detection according to the Official Mexican Standards. The objective of this work was to detect the concentrations of SO_4^{2-}, PO_4^{3-}, Ph and ClPh, present in the water for agricultural use in the areas of Xochimilco and Tlahuac. A total of 20 samples of water for agricultural use were collected, coming from 20 geographic points of the water bodies of Xochimilco and Tlahuac. The determination of SO_4^{2-}, PO_4^{3-} and total phenols was carried out by UV-VIS spectrophotometry, using the methods: molybdenum blue, modified turbidimetric, and direct spectrophotometric, respectively. Ph and ClPh were identified and quantified by a new HPLC method. Water for agricultural use presented maximum levels of SO_4^{2-}, PO_4^{3-}, total phenols and ClPh of 277.56 ± 0.3, 4.71 ± 0.004, 1.44 ± 0.01 and 0.965 ± 0.002 ppm, respectively. For water for livestock use, the maximum values were 461.03 ± 3.58, 1.71 ± 0.05, 0.78 ± 0.005 and 0.119 ± 0.004 ppm, respectively. The water for agricultural use in Xochimilco and Tlahuac has concentrations high of SO_4^{2-}, PO_4^{3-}, Ph, and ClPh. In some cases, it exceeds the established limits by Mexican regulation. Therefore, it is considered unsuitable for these activities since it represents a risk for the environment, animal health, and human health.

Key words: chlorophenol, phenol, phosphates, sulfates, water for agricultural use.

1. Introduction

Agricultural and animal production are the main source of pollution of water bodies due to the excessive use of fertilizers and the poor management of organic waste from animals (1, 2, 3). In the last three decades, concern has increased about the production, use, and destination of numerous chemical products used in industry, agriculture, livestock, among others. Some of them, such as agrochemicals, particularly pesticides, are intentionally spread over vast regions to protect different types of crops from pests. Others, such as industrial waste, are discharged directly or indirectly into water or air (4).

Agricultural activities use 77% of the water in Mexico. The regions of Xochimilco and Tlahuac in Mexico City, whit 442,178 and 392,313 inhabitants, respectively (5), are characterized by their crops of nopal, grain corn, sorghum, beans, soybeans, wheat, rice, and vegetables such as chili, potato, onion, tomato, and green tomato, among others. These regions produce 10,000 tons of vegetables annually to supply Mexico City. The rearing of cattle, pigs, sheep, and calves for fattening, equines, and poultry is also carried out (6). The primary livestock activity is milk production, followed by pork, and finally beef (7).

Xochimilco canals receive semi-purified water from industrial, domestic sources (8), productive activities, and fertilizer residues (9). These localities use it due to proximity and lack of liquid (10, 11). Water used for agricultural irrigation can contain up to 70,000 chemical compounds (12, 13), such as sulfates (SO_4^{2-}), phosphates (PO_4^{3-}), phenols (Ph), and 2-chlorophenol (ClPh).

Sulfates (SO_4^{2-}) are very abundant in nature, and their presence in water is varied. A dose above 250 ppm imparts a bitter taste to the water and causes corrosion in pipes (14, 15). It causes laxative and dehydration effects, gastrointestinal irritation in children older adults, and young cattle (16, 17). Mexican regulation in 1989 established a permitted limit of 130 ppm for agricultural irrigation water (18).

Phosphorus (P) is a chemical element widely used in the detergent industry, mostly present in the form of phosphates (PO_4^{3-}), some of them can act as nutrients for some aquatic microorganisms causing an imbalance. A content greater than 0.5 ppm of PO_4^{3-} triggers a series of events that lead to a eutrophication process (17, 19, 20, 21). Mexican legislation establishes permitted limits of 0.05 and 5 ppm (since 2009 and 1996, respectively), for waters from agroindustrial activities and the protection of aquatic life, respectively (22, 23).

Phenolic compounds (CPh) are other contaminants, such as phenol (Ph) and 2-chlorophenol (ClPh). ClPh are toxic at concentrations less than 1 μg/L, as they inactivate vital cellular proteins, including enzymes (24, 25). CPh in water can cause damage to aquatic communities and populations (26). In Mexico, since 2009, the Comisión Nacional del Agua established a maximum concentration of ClPh of 0.04 ppm and Phs of 0.1 ppm in wetlands, to protect aquatic life (22). Environmental legislation established, in 2003, maximum permissible levels for Phs (or CPh) of 0.3 ppm, in wastewater destined for the artificial recharge of aquifers (27).

The sustainable production of agricultural food demands clean technologies that avoid the excessive use of non-degradable chemicals product. Therefore, it is necessary to use sensitive analytical methods to detect these contaminants, according to the permissible limits established by the Official Mexican Standards. The objective of this

work was to determine the concentrations of SO_4^{2-}, PO_4^{3-}, Ph, and ClPh, present in the water for agricultural use in the areas of Xochimilco and Tlahuac, since these still in low concentrations, represent a risk to human, animal health and the environment.

2. Materials and Methods

2.1 Samples

A total of 20 samples of water for irrigation and animal consumption were collected, coming from 20 geographical points (in triplicate) of the water bodies of Xochimilco and Tlahuac, following national regulations (28). The samplings were in June and September (Table 1) due to the production by sowing by irrigation.

Table 1. Xochimilco and Tlahuac water bodies sampling sites.

Code	Site	Location	Use
1-3	Lagoon waterers (19°16'30.6"N 99°03'38.1"W)	Xochimilco	Livestock
4	Rancho 3 Amigos (19°13'41.9"N 99°07'02.9"W)		
5	Paddock provider (19°15'26.0"N 99°03'44.1"W)		
6	Alfalfa paddock (19°15'32.1"N 99°03'42.3"W)		
7	Cuenca lechera (19°17'04.2"N 99°04'12.0"W)		
8	Laguna (19°16'10.2"N+99°03'43.8"W)		
9	Near the Casa de la Cultura San Gregorio Atlapulco (19°15'21.9"N 99°03'20.9"W)		Agricultural
10	Supply to irrigation (19°15'22.1"N 99°03'23.6"W)		
11	Canal Nacional 13 (19°15'22.4"N 99°03'26.9"W)		
12	Chinampera zone of vegetables (19°15'50.6"N 99°02'53.7"W)		
13	Irrigation of vegetables (19°15'50.2"N 99°02'52.7"W)		
14	Near washing places (19°15'24.2"N 99°03'37.5"W)		
15-20	La Ciénega (19°16'43.0"N 99°00'37.8"W)	Tlahuac	

Samples were kept refrigerated and filtered (0.20 µ Millex® GN nylon membrane) prior to analysis to remove interferences from suspended solids. All determinations were made in triplicate, carrying out statistical analysis with a one-way ANOVA and the difference of means with a Tuckey test at 0.95%.

2.2 Determination of phosphates

For the determination of phosphates was used the molybdenum blue method (29). The standard for the calibration curve (0.0625 to 10 ppm) was KH_2PO_4 (MEYER). First, was transferred 1 mL of standard (or sample) into a 2 mL microtube (Eppendorf). After while stirring 100 µL of ammonium molybdate and 60 µL of ascorbic acid were added. Finally, the absorbance was read against a blank at 660 nm, in a VARIAN UV-VIS spectrophotometer.

2.3 Determination of sulfates

The determination of sulfates was carried out by the modified turbidimetric method (30), using Na_2SO_4 (MEYER) as a standard for the calibration curve (2.5 to 200 ppm). First, was transferred 1 mL of standard or sample to a 2

mL microtube (Eppendorf). Then, was added 50 µL of conditioning solution, stirred, and immediately a pinch of $BaCl_2$ crystals was deposited on it. It vortexed, allowed to stand for 5 min. Finally, it was read in a spectrophotometer at 420 nm against a calibration blank.

2.4 Determination of total phenols

The determination of total phenols was made with the direct spectrophotometric method (31) from a calibration curve of phenol (Sigma) as standard (0.05-10 ppm). First, was transferred 1 mL of standard or sample into a 2 mL microtube (Eppendorf). Then were added 25 µL of 0.5 N ammonia, 10 µL of 4-aminoantipyrine (2% w/v), and 10 µL of potassium ferrocyanide (4% w/v). The mixture was stirred and allowed to stand for 15 min. Finally, the absorbance was read against a blank at 510 nm in a VARIAN UV-VIS spectrophotometer.

2.5 Determination of phenol and chlorophenol by HPLC

2.5.1 Operating conditions

Equipment: Varian ProStar 325 High-Resolution Liquid Chromatograph, Nucleosil 100-5 C18 125x4.6 mm column (720002.46), pump model PS-230, UV-Vis detector of variable wavelength model PS-325, manual injector and Galaxie Workstation software version 1.9 SP2. Operating conditions: mobile phase acetonitrile/water (80:20 v/v), the flow of 0.5 mL/min, injection volume of 10 µL and 268 nm. The standards were Ph and ClPh HPLC grade (Sigma Aldrich).

2.5.2. Method validation

The method was validated, according to the Analytical Method Validation Guide (32).

System precision. Phenol and chlorophenol solutions were prepared five-fold, at 1 ppm. The analytical response was measured by HPLC, with the operating conditions described above. The acceptance criterion for the precision of the system was CV≤3%.

System adequacy. A solution of Ph and ClPh at 1 ppm, separately with the same operating conditions, was injected into the HPLC five times.

Linearity of the method. Phenol and chlorophenol solutions were prepared, at a concentration of 10 ppm. Subsequently, were carried out the calibration curves at concentrations of 0.01 to 6 ppm. The acceptance criterion was a determination coefficient (r^2) ≥ 0.98.

3. Results and Discussion

3.1 Determination of sulfates

50 % of the analyzed samples presented SO_4^{2-} concentrations above 130 ppm, and 25% were above 250 ppm. These simples come from the lagoon water trouh, the chinampera area of Xochimilco, and the Tlahuac Cienega (Figure 1).

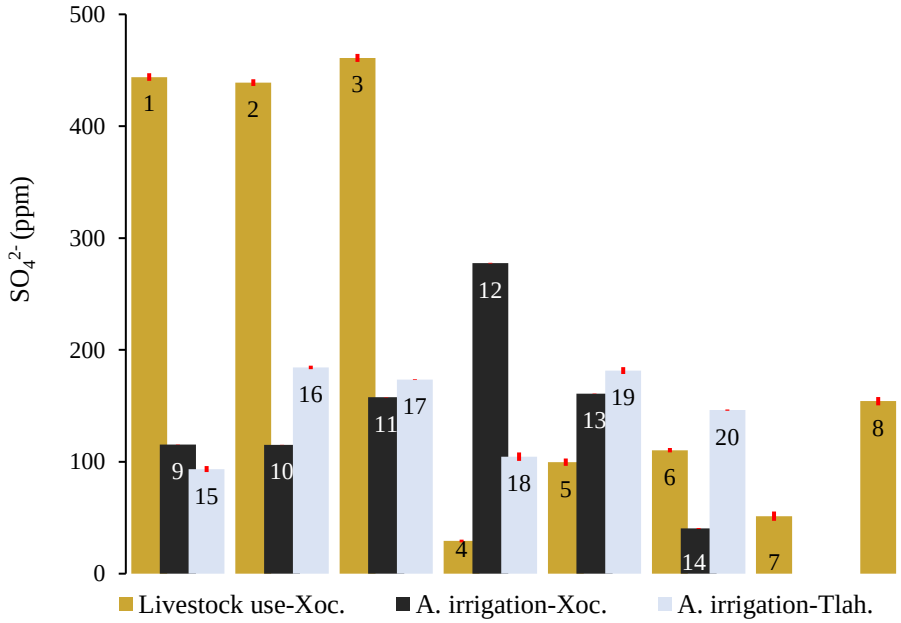


Figure 1. SO_4^{2-} in samples of water for agricultural use in Xochimilco and Tlahuac.

These concentrations are higher than the reported by Arana for the Xochimilco area of 214, 79, and 63 ppm (33, 34). They also exceed the limits allowed by the Ecological Criterion (18) and the Federal Law on Water Rights (22) for water from irrigation of food and vegetables for raw consumption (130 ppm) and as an animal drink (250 ppm). The Tlahuac Cienega, considered a Ramsar wetland, is a vital aquatic environment for a great diversity of local and migratory birds, among others (35). These results are relevant since they suggest excessive use of agrochemicals and pesticides, especially nitrogenous and phosphates, to guarantee harvests. According to SIAP-SAGARPA, in 2015, between 74 and 95% of the sown area was fertilized with chemicals (36). However, sulfates in high concentrations can affect animal health and potentially human health (37, 38). They can have different degrees of toxicity to aquatic biota, depending on time depth and urban discharges (39). In children, they cause a laxative effect (40) and salinity problems in irrigation water, foliar necrosis, crop reduction, and loss of germination capacity, among other damages (41).

3.2 Determination of phosphates

The PO_4^{3-} concentrations in the samples from Xochimilco were higher, in contrast to those from the Tlahuac zone, highlighting the irrigation water from the chinampera zone (Figure 2). In all cases, was exceeded the limit for waters from agroindustrial activities of 0.05 ppm (22), and the limit of 5 ppm for aquatic life protection (23) was below. However, small concentrations of PO_4^{3-} (0.5 ppm) damage ecosystems, safety, and food production for human consumption (19, 21, 42). In this sense, 65% of the analyzed samples presented PO_4^{3-} levels above 0.5 ppm.

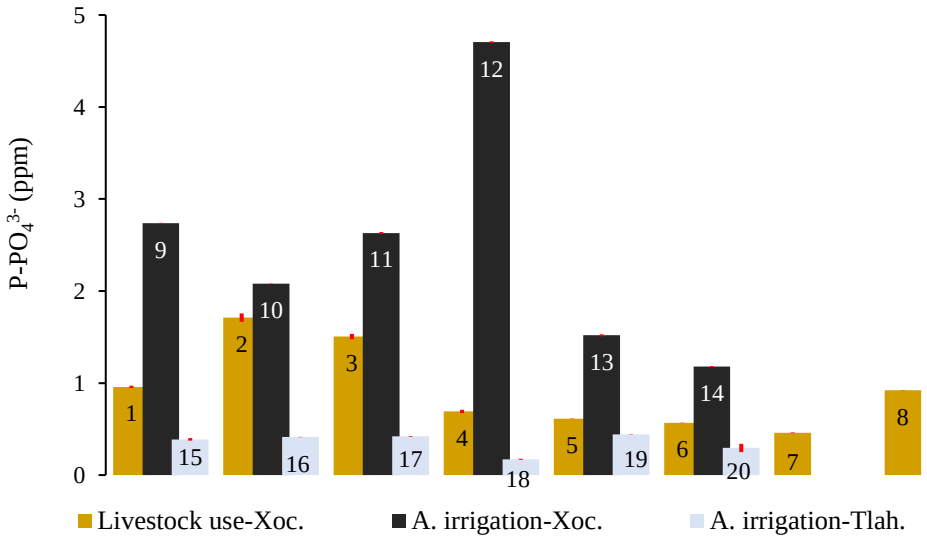


Figure 2. PO_4^{3-} in samples of water for agricultural use in Xochimilco and Tlahuac.

The phosphate concentrations found in this study were lower than that reported by some authors; Arana et al. reported in 2006 and 2009, 11.3 and 8.1 ppm, respectively (33, 34). Zambrano et al. (2009) quantified 7.2 ppm in the chinampera zone (43). However, Moreno-Franco et al. in 2010 (44) and Zarazúa et al. in 2103 (45) reported that concentrations between 1.177 and 0.294 ppm of PO_4^{3-} indicate eutrophic conditions. Pérez-Díaz et al., in 2019, described that a value of 2.35 ppm indicates a hypereutrophic state (46). They coincided with Ferrara et al. (2014) in that the Xochimilco canals are eutrophic in the dry season and hypereutrophic in the rainy season (47). Similarly, the Tlahuac Cienega is a body eutrophic by the discharges of agricultural and domestic wastewater that contain inorganic nitrogen and phosphorous compounds (48), and excessive use of fertilizers (49). In this sense, 80% of the analyzed sites presented eutrophic conditions and 15% hypereutrophic conditions.

It is interesting to mention the excessive use of household detergents. Detergents used in dairy production facilities such as the Cuenca Lechera of Xochimilco, among others, can be accidentally dumped into canals or rivers, causing contamination. In some European countries are prohibited detergents containing phosphates and sulphates because they are not easily degradable compounds. Their use has impacted the quality of rivers and lakes and the imbalance in aquatic life (50). These effects are still present in some countries such as Europe, Japan, and the United States (51, 52).

3.3 Determination of total phenols

All samples exceeded the phenol limit (Figure 3) of 0.1 ppm in wetlands to protect aquatic life (22). 75% of the samples had concentrations above 0.3 ppm, the limit allowed in wastewater destined for the artificial recharge of aquifers (27). The black water sample (which goes to the canal) and two of animal consumption (simples 2 and 8) from the Xochimilco area stood out.

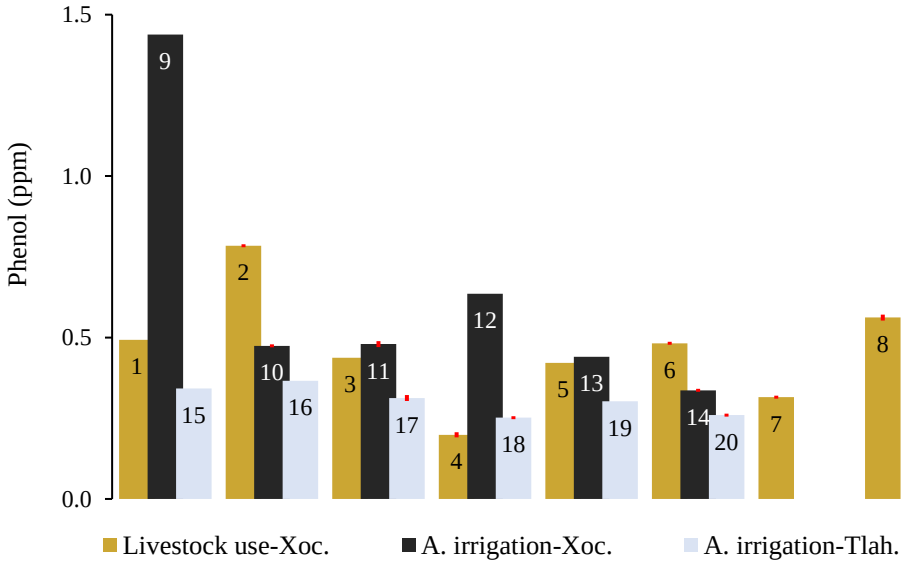


Figure 3. Phenol in samples of water for agricultural use in Xochimilco and Tlahuac.

The presence of phenolic compounds in these bodies of water can provocate color changes and impart a bad smell for the oxidation of said compounds (53). Some phenols are used as pesticides, causing their diffusion through agricultural activities and entry into the food chain (54). They can impart a bitter taste to aquatic species for human consumption. In living beings, it affects the liver, kidneys, lungs, and vascular system. Long-term exposure can lead to coma, seizures, cyanosis, and even death (53). Therefore, it is relevant to monitor and control these pollutants in water for agricultural use to avoid health problems, mainly for farmers and inhabitants of those areas.

3.4 Determination of phenol and chlorophenol by HPLC

The spectrophotometric technique does not allow to differentiate between the different phenols present in the simples. Its toxicity varies according to the type of phenol present, like phenol, nitrophenol, and chlorophenol. For this reason, a method to determine the presence of phenol, and chlorophenol in some samples, was established.

The established conditions were the most suitable for the determination of phenol and chlorophenol, allowing the identification and separation of both compounds. Retention times were 2.7 ± 0.71 and 2.9 ± 0.75 min, respectively, with a run time of 5 min.

The method complies with the established validation parameters. The detection limits for Ph and ClPh were 0.02 and 0.01 ppm, respectively (Figure 4). The methodology is specific to identifying and quantifying phenolic compounds. The presence of other compounds does not interfere with the analysis.

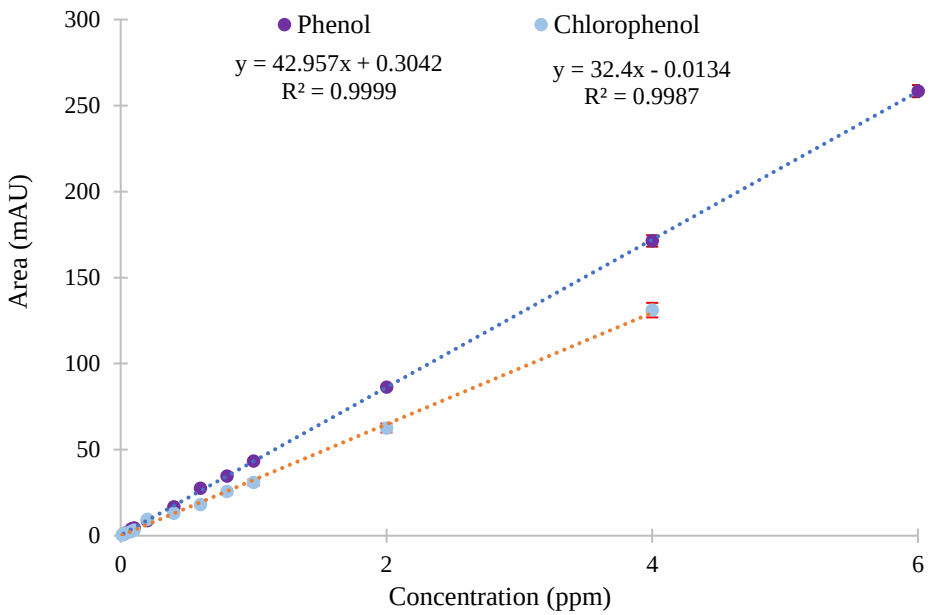


Figure 4. Calibration curve for Ph and ClPh.

The replications in the determinations of Ph and ClPh at 1 ppm (Table 2) had a precision interval of 2.28 and 2.70%, respectively. Therefore, the accuracy of the system met the established acceptance criteria.

Table 2. System accuracy with 1 ppm phenol and chlorophenol standard solution.

Phenol		**Chlorophenol**	
Retention time (min)	Area (mUA)	Retention time (min)	Area (mUA)
2.73	44.56	2.88	29.88
2.68	43.67	2.89	30.05
2.71	42.78	2.93	30.25
2.69	44.79	2.92	29.55
2.70	45.35	2.88	29.35
Prom	44.23		30.216
Desvest	1.012		0.817
C.variation	2.287		2.704

The method used had several advantages compared to others, such as the spectrophotometric with color development, the extraction with chloroform, and the gas chromatography. It was faster, more accurate, and with detection limits below those allowed by Mexican regulation. It can be applied in water samples and is comparable with the method of Silva et al. (54).

Once the method was validated, only the water effluent samples from livestock and agricultural activities in the Xochimilco area were analyzed, which yielded the highest concentrations of total phenols by spectrophotometry. The samples without previous treatment were analyzed, which optimized the analysis times. However, according to Nieves et al. in 2015, when are pre-concentrated with the solid phase extraction technique, units of parts per million can be reached (55).

The samples of water for livestock use presented higher levels of Ph. In contrast, the water samples for agricultural use had higher levels of ClPh (Figure 5). These results are consistent with those found by spectrophotometry for total phenols in this study.

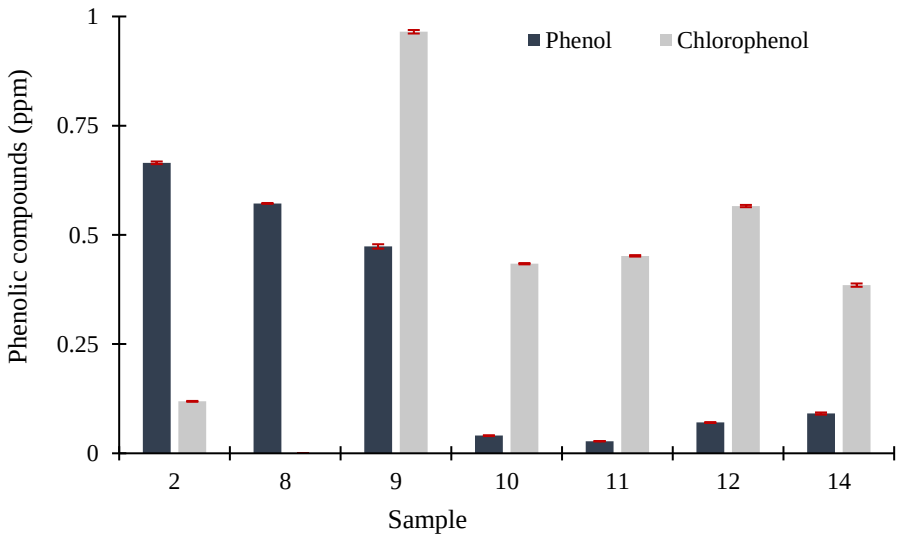


Figure 5. Ph and ClPh in samples of livestock water (2 and 8) and agricultural use in Xochimilco.

In agricultural activity, is used many pesticides that contain chlorinated phenols and nitrophenols. These are very toxic to aquatic life, as is the case of chlorophenols at concentrations less than 1 µg/L (25). According to the Mexican Regulation, 57% of the samples were above the limits allowed for ClPh, 42.8% were above the limits for Ph (22), and 57% presented concentrations higher than the permitted limits for phenolic compounds (27).

In the environment, these compounds, at these concentrations, can have serious repercussions, especially in aquatic ecosystems and in human (56) and animal health. Some studies indicate that the consumption of water contaminated with these compounds causes nausea, diarrhea, and headache (57). According to the health statistics of these areas, gastrointestinal diseases are one of the most common conditions, and children are the most vulnerable to them. In animals, it causes damage to the kidneys, liver, and gastrointestinal, circulatory, and respiratory systems (58).

4. Conclusions

The water for agricultural use in Xochimilco and Tlahuac has concentrations high of sulfates, phosphates, phenol, and chlorophenol. In some cases, it exceeds the established limits by Mexican regulation. Therefore, it is considered unsuitable for these activities, representing a risk for the environment, animal health, and human health.

The proposed method for the determination of phenol and chlorophenol by HPLC with UV detector is fast, sensitive, and applicable to water samples, with detection limits for maximum permissible values established by national and international regulations.

It is necessary to carry out periodic monitoring of water for agricultural use in the different seasons of the year, in the primary production of agricultural foods, and comply with the regulatory framework on the limits of chemical substances allowed for these activities.

5. Acknowledgements

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