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МЕТОД ВЭЖХ ДЛЯ ОПРЕДЕЛЕНИЯ ОСТАТКОВ НЕКОТОРЫХ АНТИБИОТИКОВ В СТОЧНЫХ ВОДАХ РАЗЛИЧНЫХ БОЛЬНИЦ В ГОРОДЕ БАГДАД, ИРАК

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Антибиотики представляют наибольшую угрозу для почвы и водных экосистем среди различных терапевтических групп лекарств (которые включают отпускаемые по рецепту лекарства и средства для лечения рака). Сильнейшие лекарства – антибиотики – использовались для остановки роста микроорганизмов или их уничтожения. С помощью технологии высокоэффективной жидкостной хроматографии с флуоресцентным детектированием были определены количества левофлоксацина и тетрациклина в сточных водах трех больниц (Медикал Сити, Аль-Кинди и Аль-Ярмук). В этом исследовании были выбраны левофлоксацин и тетрациклин, поскольку они являются наиболее важными загрязнителями воды. Эти остатки антибиотиков были разделены и измерены с использованием метода градиентного элюирования на колонке с обращенной фазой С18. Комбинация метанола и деионизированной воды составляла систему подвижной фазы. Для 20-минутного периода анализа длины волн возбуждения и излучения детектора были установлены на 310 и 420 нм соответственно. Очистку картриджа ТФЭ проводили после экстракции с использованием цитратного буфера с pH 4. Прекрасные линейные зависимости (R^2 > 0,9998) наблюдались на калибровочных кривых для левофлоксацина и тетрациклина в концентрациях от 10 до 40 мкг/мл. Пределы обнаружения и количественного определения левофлоксацина и тетрациклина составили 0,61 мкг/мл, 2,04 мкг/мл, 0,46 мкг/мл и 1,54 мкг/мл соответственно. С использованием предложенной методики был успешно применен анализ остатков антибиотиков в различных пробах сточных вод. Результаты показывают наличие тетрациклина и левофлоксацина во всех пробах сточных вод. Однако в Аль-Кинди их концентрация была выше, чем в Медикал-Сити и Аль-Ярмуке. Предложенный метод может быть применен к ряду медицинских продуктов в различных источниках сточных вод, таких как больницы и промышленные предприятия.

Ключевые слова: остатки антибиотиков, ВЭЖХ, левофлоксацин, тетрациклин, сточные воды

HPLC METHOD FOR THE DETERMINATION OF SOME ANTIBIOTIC RESIDUES IN DIFFERENT HOSPITALS WASTEWATER IN BAGHDAD CITY, IRAQ

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Antibiotics present the greatest threat to soil and aquatic ecosystems among the different therapeutic groups of medicines (which include prescription drugs and treatments for cancer). The

strongest drugs, antibiotics, have been utilized to stop the growth of microorganisms or eradicate them. Using high-performance liquid chromatography technology with fluorescence detection, the amounts of levofloxacin and tetracycline in the wastewater from three hospitals (Medical City, Al-Kindi, and Al-Yarmouk) were determined. Levofloxacin and tetracycline were chosen in this study because they are the most important water pollutants. These antibiotic residues were separated and measured using a gradient elution technique on a reverse-phase C18 column. A combination of methanol and deionized water made up the mobile phase system. For a 20-minute analysis period, the detector's excitation and emission wavelengths were set to 310 and 420 nm, respectively. Cleaning the SPE cartridge came after the extraction using pH 4 citrate buffer. Excellent linear ties (R^2 > 0.9998) were seen in the calibration curves for levofloxacin and tetracycline at concentrations between 10 and 40 μg/ml. The limits of detection and quantification for levofloxacin and tetracycline were determined to be 0.61 μg/ml, 2.04 μg/ml, and 0.46 μg/ml and 1.54 μg/ml, respectively. The analysis of residues of antibiotics in various wastewater samples was successfully applied using the suggested methodology. The findings demonstrate the presence of tetracycline and levofloxacin in all wastewater samples. However, Al-Kindi had greater concentrations of them than Medical City and Al-Yarmouk. The proposed technique can be applied to a range of medical products in different wastewater sources, such as hospitals and industrial settings.

Keywords: antibiotic residues, HPLC, levofloxacin, tetracycline, wastewater

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INTRODUCTION

All areas of medical facilities, including hospitals, typically produce hospital effluents are a substantial source of new pollutants in many developing nations. Among these pollutants include heavy metals [1], genes for antibiotic resistance, microbes resistant to antibiotics, and antibiotic residues. Since pathogenic bacteria have developed antibiotic resistance mechanisms over time, they have long been able to successfully oppose them [2]. Antibiotics have been used extensively worldwide to cure or prevent bacterial illnesses as well as to aid in animal growth [3-8]. According to reports, the usage of antibiotics by humans worldwide climbed by 65% between 2000 and 2015, and if the current pattern of consumption continues, it may increase by 200% in 2030. The major threat to soil and aquatic ecosystems from the various therapeutic groups of medicines (such as medications and anticancer treatments,) is from antibiotics [9]. Antibiotics are the most powerful medications and have been used to either kill or stop the growth of microorganisms [10]. Antibiotics work in a variety of ways, one of which is to prevent the formation of peptidoglycans and nucleic acids, which has a detrimental effect on cell division and ultimately causes cell death [11].

Conventional water purification methods generate hazardous waste and byproducts, necessitating energy-intensive processing. A significant overhaul is needed for sustainable development and green technologies [12]. Environmentalists have recently faced a conundrum as a result of the finding of recent contaminants in various surface water bodies all over the world, such as pharmaceutical and personal care products, polycyclic aromatic hydrocarbons [13], and phenols [14]. The manufacture and consumption of pharmaceutical products have increased dramatically in recent decades due to medical breakthroughs [15]. The necessary rise in their use and increasing presence in various environmental components has led to the classification of pharmaceutically compounds as emerging environmental pollutants [16]. Many techniques for identifying the key members of this class in a variety of Due to the widespread problem of drug pollution and environmental risk assessment, environmental samples were examined [17]. Pharmaceutical compounds are categorized as emerging contaminants in wastewater and biological degradation environments due to the lack of environmental discharge limits and the effects they have on the environment [18-21]. Wastewater treatment plant (WWTP) discharges are a

significant source of antibiotics that end up in environmental waterways [22-25]. These discharges have an immediate impact on the receiving water body [26, 27]. Compared KrCl-excilamps and XeBr-excilamps for simultaneous treatment and disinfection of model aqueous solutions, finding KrCl excilamp was more efficient for direct photolysis and oxidation in the peroxide system [28].

The use of advanced methods of analysis is essential for monitoring antibiotics in wastewater [29, 30], assessing potential problems, evaluating treatment plant efficiency, monitoring tertiary systems, performing environmental risk assessments, and estimating population consumption using a wastewater-based epidemiology approach, both in influent and effluent samples [26, 31-34]. The most popular technique at present is liquid chromatography connected to tandem mass spectrometry (LC-MS/MS) due to its strong potential for identification and quantification, high selectivity and sensitivity, and robustness [35-39]. The creation of analytical techniques for the sensitive and focused detection and measurement of antibiotic contamination in the environment is required [40]. Spectrophotometric techniques can help determine environmental and pharmaceutical samples because they are straightforward and affordable [41-43]. In addition, ionization mass spectrometry has been used to examine a wide variety of compounds, including small pharmaceutical compounds [44].

Tetracycline is frequently utilized antibacterial substance in both human and veterinary settings. They could be semi-synthetic or organically occurring. Tetracycline is very efficient against a wide range of bacteria. By attaching to the 30S component of the bacterial ribosome, their mechanism of action involves inhibiting protein synthesis [45]. Levofloxacin sometimes known as -ofloxacin, is a racemic fluoroquinolone antibiotic from the third generation. Since DNA gyrase is inhibited, which is essential for DNA replication and causes bacterial lysis, it has wide-ranging bactericidal effects on both Gram-positive and Gram-negative bacteria [46].

Tetracycline antibiotics were found to be the most significant water pollutants among those prescribed in Iraqi drug enterprises because of their extensive use and weak to moderate adsorption to sediments [47]. A chromatographic technique was used to determine the levels of tetracycline in water samples from environmental sources. Samples were acidified, purified, and then re-dissolved using a solution of methanol and oxalic acid. Tetracycline linearity was determined using HPLC-DAD [48]. In the study by Xin and coworkers, tetracycline was initially extracted from environmental water samples using a green extractant, hydrophobic deep eutectic solvent, to be determined by HPLC [49]. Tetracycline was also detected with extreme sensitivity using a Raman fingerprint technique. The detection limit for tetracycline standard solutions was 0.04 ng/mL, and the amounts found ranged from 0.5 to 50 ng/ml [50]. Levofloxacin is a bacteriostatic because it is an antibiotic with a broad spectrum that inhibits bacterial growth [51, 52]. Levofloxacin residues have been detected in aqueous samples using a variety of techniques, including solid-phase microextraction, dispersive-solid phase extraction, molecularly imprinted polymer, and other methods based on microextraction principles [53].

The yields in this study varied from those in other experiments. The majority of previous studies have simply looked at the methods for detecting tetracycline and levofloxacin in human plasma and aqueous samples, without further developing or researching them, such as the methods for detecting these antibiotics in hospital wastewater. The specific objective of this study was to offer a rapid, inexpensive, and reliable analytical method for levofloxacin and tetracycline determination in wastewater from three hospitals (Medical City, Al-Kindi, and Al-Yarmouk).

MATERIAL AND METHODS

Chemicals and reagents

Levofloxacin and tetracycline standards, as well as phosphate buffer (Na_2HPO_4), were supplied by the German company Sigma-Aldrich. Methanol was HPLC-grade which was provided by Biosolve company, France.

By utilizing a Strata C_{18}-E cartridge (500 mg, 6 ml; Phenomenex, Milford, MA, USA), the extracts were cleaned using solid phase extraction.

Apparatus

Analysis was done using an HPLC (model SYKAM – German) and fluorescence detector with excitation wavelength (Ex) = 310 nm and emission wavelength (Em) = 420 nm and ODS C_{18} column (250 mm 4.6 mm 5 μm).

Preparation of standards

High-quality methanol was used to dissolve 0.2 mg of each standard compound in a 5 ml volumetric flask to prepare 40 μg/ ml of stock solution. After that, several standard solutions were prepared (10, 20, 30, and 40 μg/ ml).

Preparation of samples and extraction procedure

500 ml of each sample was collected in clean polyethylene terephthalate bottles from the wastewater of three hospitals (Medical City, Al-Kindi, and Al-Yarmouk). A high-speed tissue mixer was used to combine each sample. 10 ml of citrate buffer (7:3) and methanol (7:3) v/v were added. At room temperature, the mixture was vortexed for 5 min. 10, in a centrifuge that had been cooled, at 3,500 rpm. It was possible to repeat the extraction by adding 2 ml of citrate buffer. The supernatant was preconditioned with 10 ml of methanol and 4 ml of water before being filtered and placed onto an SPE cartridge. Before the tetracyclines were extracted, the sample-containing cartridge was washed with 5 ml of water. Then, 3 mL of the eluent was filtered through a 0.45-m nylon filter. The HPLC system received 100 L of the aliquot.

HPLC conditions

Gradient elution was carried out utilizing a methanol and deionized water mixture (70:30 v/v). For the chromatographic analysis, a reversed-phase column (4.6 mm, 250 mm, µ5 m) was employed. The column was preheated to 35 °C, and 100 µL of injection was use.

RESULTS AND DISCUSSION

The suggested method

This study used HPLC, a more advanced chromatographic method, to analyze levofloxacin and tetracycline in wastewater from three hospitals (Medical City, Al-Kindi, and Al-Yarmouk). HPLC provides several advantages over other chromatographic methods, including significantly better sensitivity and resolution as well as the ability to do quantitative analysis. The separation conditions were carried out in a gradient elution of methanol and deionized water. The method involves extraction of the samples from wastewater and clean-up on solid phase extraction. A ODS C18 column is used to separate the samples at a flow rate of 1.2 ml/min. The HPLC findings demonstrated that the technique can quickly identify the standard materials based on the peak retention times in the chromatogram.

Identification of standards

Each standard solution (both 10 µg/ ml) of levofloxacin and tetracycline was injected into the HPLC system using the same conditions. The retention time for levofloxacin is 6.98 min (Fig. 1), whereas the peak for tetracycline is 4.08 min (Fig. 2). The peak area and the peak height were also calculated and found to be 1958.79 mAU, 830.14 mAU for levofloxacin and 1524.49 mAU, 823.65 mAU for tetracycline.

Method validation

The linearity response of each drug was measured by plotting the drug peak area versus the drug concentration, and the linearity response was used to develop the regression equations. The calibration curve for each drug was linear and covered a range of 10-40 µg /mL, Fig. 3 and Fig. 4. For each drug, the correlation coefficients (R^2) were greater than 0.9998, indicating that the method is linear within the specified range. The findings demonstrated that a trace amount of a component can be found in pharmaceutical mixtures using HPLC, utilizing either of the studied sampling techniques. Utilizing the slope and standard deviation, Calculations were made to determine the limits of detection (LOD) and quantification (LOQ):

$$LOD = \frac{STEYX}{Slope} \times 3$$

$$LOQ = \frac{STEYX}{Slope} \times 10$$

STEYX means the standard deviation of the y-value and x-value.

It could be seen that the suggested method's LOD and LOQ for levofloxacin and tetracycline, respectively, were 0.61 µg/ ml, 2.04 µg/ ml, and 0.46 µg/ ml and 1.54 µg/ ml, respectively. The obtained results from the analysis of levofloxacin and tetracycline are summarized in Table 1.

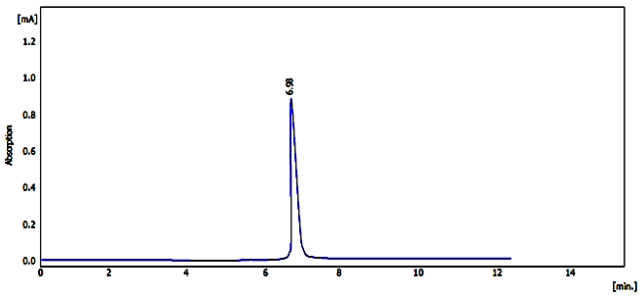

Fig. 1. HPLC chromatogram for levofloxacin (10 µg/ml)
Рис. 1. ВЭЖХ-хроматограмма левофлоксацина (10 мкг/мл)

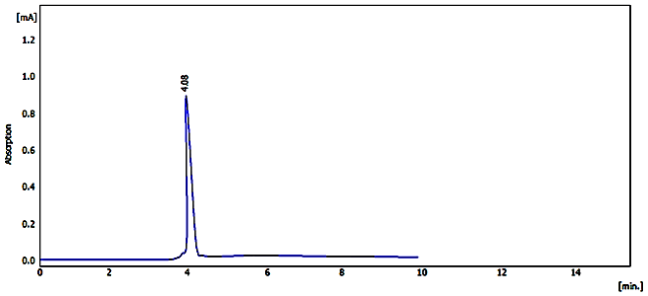

Fig. 2. HPLC chromatogram for tetracycline (10 µg/ml)
Рис. 2. ВЭЖХ-хроматограмма тетрациклина (10 мкг/мл)

Table 1

Analytical characteristics of the suggested method

***Таблица 1*. Аналитические характеристики предлагаемого метода**

Standard	Linear range (μg/ml)	Regression equation	Correlation coefficients (R^2)	LOD (μg/ ml)	LOQ (μg/ ml)
Levofloxacin	10-40	y = 197.47x	0.9999	0.61	2.04
Tetracycline	10-40	y = 154.75x	0.9999	0.46	1.54

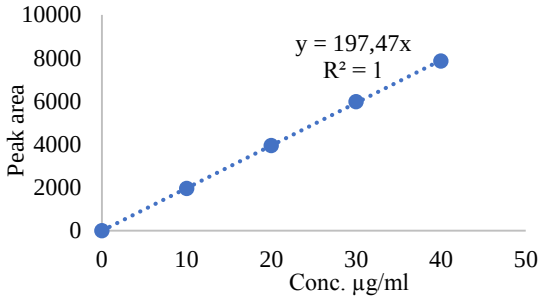

Fig. 3. Calibration curve of levofloxacin
Рис. 3. Калибровочная кривая левофлоксацина

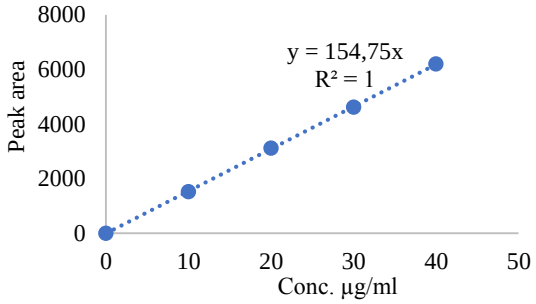

Fig. 4. Calibration curve of tetracycline
Рис. 4. Калибровочная кривая тетрациклина

Method application

Local wastewater samples were analyzed for levofloxacin and tetracycline residues in three different hospitals (Medical City, Al-Kindi, and Al-Yarmouk). The findings showed that the following antibiotic residues were frequently found in local wastewater samples. Levofloxacin and tetracycline were found and measured in samples that were positive, Fig. 5-7.

The results showed that levofloxacin residues in the wastewater samples ranged from 35.20 to 42.59 μg/ml, with a mean of 38.50 μg/ml, while they showed that tetracycline residues ranged from 55.72 to 61.72 μg/ml, with a mean of 58.58 μg/ml. Tables 2, 3, and 4 show and compare the experimental data on HPLC determination of levofloxacin and tetracycline in the wastewater samples of Medical City, Al-Kindi, and Al-Yarmouk hospitals.

In summary, these results show that both antibiotics (levofloxacin and tetracycline) can be detected in all wastewater samples. However, the levels of them were higher in Al-Kindi compared to those of Medical City and Al-Yarmouk. A possible explanation for this might be the hospital is located near densely populated cities, which contributes to its large patient population in comparison with those of others.

CONCLUSION

The purpose of this work is to use HPLC-fluorescence detection and a C18 column to quickly and easily determine the presence of tetracycline and levofloxacin in wastewater samples from three hospitals: Medical City, Al-Kindi, and Al-Yarmouk. The recommended methodology was successfully used to analyze the antibiotic residues in different wastewater

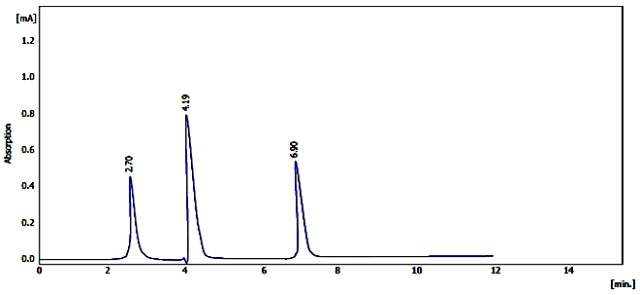

Fig. 5. Chromatogram of Medical City sample
Рис. 5. Хроматограмма образца Medical City

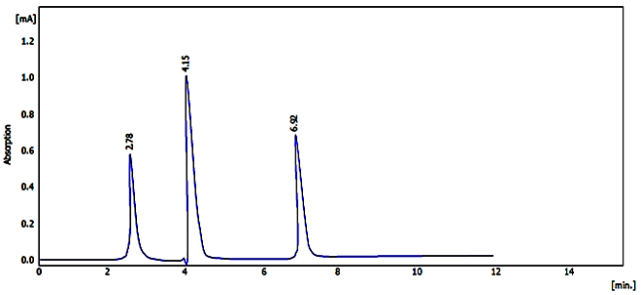

Fig. 6. Chromatogram of Al-Kindi sample
Рис. 6. Хроматограмма образца Аль-Кинди

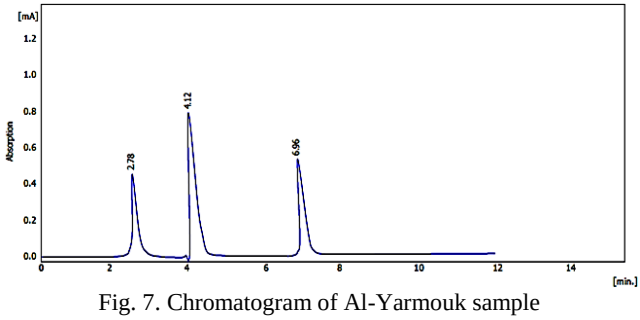

Fig. 7. Chromatogram of Al-Yarmouk sample
Рис. 7. Хроматограмма образца Аль-Ярмук

samples. As a result, it can be concluded that chromatographic and extraction techniques help identify and measure the amount of antibiotics in water. The suggested approach worked well for examining the antibiotic residues found in various wastewater samples. The results showed that levofloxacin and tetracycline were present in all wastewater samples. Al-Yarmouk and Medical City would have lower concentrations of them than Al-Kindi. The suggested method can also be used for various medical products in different wastewater sources, including hospitals and industries.

Table 2

HPLC determination of levofloxacin and tetracycline in Medical City sample

***Таблица 2.* ВЭЖХ-определение левофлоксацина и тетрациклина в образце Medical City**

Drug	Found (μg/ml)	Retention time (min)	Peak area (mAU.s)	Peak height (mAU)	Area (%)	Height (%)
Levofloxacin	35.20	6.90	6952.08	584.19	34.79	34.69
Tetracycline	55.72	4.19	8623.41	799.58	40.15	40.20

Table 3

HPLC determination of levofloxacin and tetracycline in Al-Kindi sample

***Таблица 3*. ВЭЖХ-определение левофлоксацина и тетрациклина в образце Аль-Кинди**

Drug	Found (μg/ml)	Retention time (min)	Peak area (mAU.s)	Peak height (mAU)	Area (%)	Height (%)
Levofloxacin	42.59	6.92	8412.58	735.25	34.79	34.69
Tetracycline	61.72	4.15	9541.08	988.47	40.15	40.20

Table 4

HPLC determination of levofloxacin and tetracycline in the Al-Yarmouk sample

***Таблица 4*. ВЭЖХ-определение левофлоксацина и тетрациклина в образце Аль-Ярмука**

Drug	Found (μg/ml)	Retention time (min)	Peak area (mAU.s)	Peak height (mAU)	Area (%)	Height (%)
Levofloxacin	37.73	6.96	7451.96	586.08	34.79	34.69
Tetracycline	58.31	4.12	9025.44	795.49	40.15	40.20

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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